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Ammonium Metatungstate (AMT)

Physical & Chemical Properties, Processes, & Applications

chinatungsten.com

chinatungsten.com CTIA GROUP LTD

Global Leader in Intelligent Manufacturing for Tungsten, Molybdenum, and Rare Earth Industries

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Introduction to CTIA GROUP

CTIA GROUP LTD, a wholly-owned subsidiary with independent legal personality established by CHINATUNGSTEN ONLINE, is dedicated to promoting the intelligent, integrated, and flexible design and manufacturing of tungsten and molybdenum materials in the Industrial Internet era. CHINATUNGSTEN ONLINE, founded in 1997 with www.chinatungsten.com as its starting point-China's first top-tier tungsten products website-is the country's pioneering e-commerce company focusing on the tungsten, molybdenum, and rare earth industries. Leveraging nearly three decades of deep experience in the tungsten and molybdenum fields, CTIA GROUP inherits its parent company's exceptional design and manufacturing capabilities, superior services, and global business reputation, becoming a comprehensive application solution provider in the fields of tungsten chemicals, tungsten metals, cemented carbides, high-density alloys, molybdenum, and molybdenum alloys.

Over the past 30 years, CHINATUNGSTEN ONLINE has established more than 200 multilingual tungsten and molybdenum professional websites covering more than 20 languages, with over one million pages of news, prices, and market analysis related to tungsten, molybdenum, and rare earths. Since 2013, its WeChat official account "CHINATUNGSTEN ONLINE" has published over 40,000 pieces of information, serving nearly 100,000 followers and providing free information daily to hundreds of thousands of industry professionals worldwide. With cumulative visits to its website cluster and official account reaching billions of times, it has become a recognized global and authoritative information hub for the tungsten, molybdenum, and rare earth industries, providing 24/7 multilingual news, product performance, market prices, and market trend services.

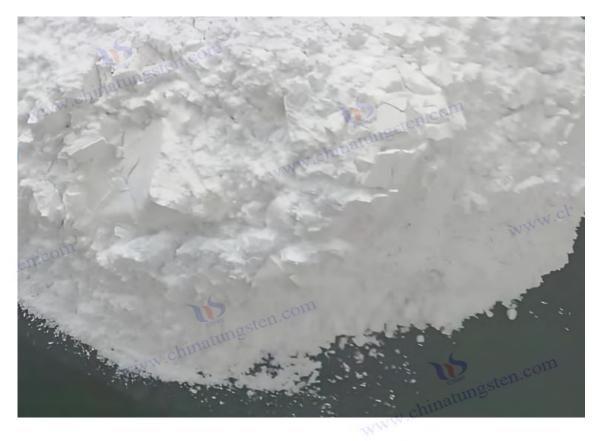
Building on the technology and experience of CHINATUNGSTEN ONLINE, CTIA GROUP focuses on meeting the personalized needs of customers. Utilizing AI technology, it collaboratively designs and produces tungsten and molybdenum products with specific chemical compositions and physical properties (such as particle size, density, hardness, strength, dimensions, and tolerances) with customers. It offers full-process integrated services ranging from mold opening, trial production, to finishing, packaging, and logistics. Over the past 30 years, CHINATUNGSTEN ONLINE has provided R&D, design, and production services for over 500,000 types of tungsten and molybdenum products to more than 130,000 customers worldwide, laying the foundation for customized, flexible, and intelligent manufacturing. Relying on this foundation, CTIA GROUP further deepens the intelligent manufacturing and integrated innovation of tungsten and molybdenum materials in the Industrial Internet era.

Dr. Hanns and his team at CTIA GROUP, based on their more than 30 years of industry experience, have also written and publicly released knowledge, technology, tungsten price and market trend analysis related to tungsten, molybdenum, and rare earths, freely sharing it with the tungsten industry. Dr. Han, with over 30 years of experience since the 1990s in the e-commerce and international trade of tungsten and molybdenum products, as well as the design and manufacturing of cemented carbides and high-density alloys, is a renowned expert in tungsten and molybdenum products both domestically and internationally. Adhering to the principle of providing professional and high-quality information to the industry, CTIA GROUP's team continuously writes technical research papers, articles, and industry reports based on production practice and market customer needs, winning widespread praise in the industry. These achievements provide solid support for CTIA GROUP's technological innovation, product promotion, and industry exchanges, propelling it to become a leader in global tungsten and molybdenum product manufacturing and information services. chinatung



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Ammonium Metatungstate (AMT) Physical & Chemical Properties, Processes, & Applications

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Ammonium metatungstate product introduction

1. Product Overview

Ammonium metatungstate (AMT) with the chemical formula (NH₄)₆H₂W₁₂O₄₀·xH₂O, is a highly soluble tungsten compound with a white or yellowish crystalline powder. AMT is an important intermediate raw material for the production of tungsten products and other tungsten compounds, and is widely used in many industrial fields due to its excellent water solubility (solubility up to 303.9g/100g H₂O at 20°C) and thermal stability.

Second, product characteristics

Appearance: White or yellowish crystalline powder

Purity: ≥99.95%

Solubility: High water solubility, insoluble in ethanol

Density: approx. 2.3 g/cm³

Thermal stability: decomposes into tungsten trioxide (WO₃) above 300°C

Safety: It is slightly acidic and irritating, so you need to pay attention to protection when using it tungsten.

3. Product specifications

WO3 含量	(≥% mi	n) 91.0		WW. Child						
Impurity content (max., %)										
element	To the	As	Bi	Ca	With	Fe	Mg	Κ	Mn	Мо
maximum	0.0010	0.0010	0.0001	0.0010	0.0005	0.0020	0.0005	0.0010	0.0010	0.0030
element	On	Nor	Р	Pb	S	Sb	Yes	Sn	Ti	V
maximum	0.0020	0.0005	0.0007	0.0010	0.0030	0.0005	0.0010	0.0010	0.0010	0.0010

4. Packaging and warranty

Packing: Inner sealed vacuum plastic bag, outer iron drum or plastic drum, net weight 50kg, moisture-proof and anti-oxidation.

Warranty: With quality certificate, tungsten content, impurity analysis (ICP-MS), particle size (FSSS method), loose density and moisture data, shelf life of 12 months (sealed and dry conditions).

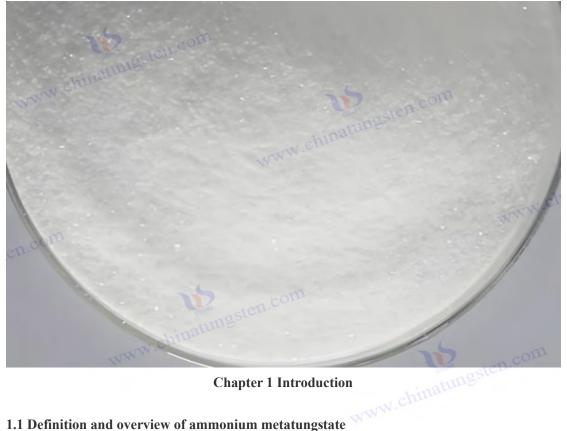
5. Procurement information

Mailbox:sales@chinatungsten.com Phone: +86 592 5129696

For more information about ammonium metatungstate, please visit China Tungsten Online (www.ammonium-metatungstate.com). www.chinatungsten

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Chapter 1 Introduction

1.1 Definition and overview of ammonium metatungstate

Ammonium metatungstate (AMT, chemical formula (NH4)6H2W12O40 nH2O) is an important polytungstate compound, as a key intermediate in the tungsten chemical industry chain, it has attracted attention for its excellent chemical and physical properties. Its molecular structure consists of a Keggin-type polyacid anion [H₂W₁₂O₄₀]⁶⁻ and 6 ammonium cations (NH₄+The amount of crystallized water (n) usually varies between 3-6, depending on the preparation conditions. Significant properties of AMT include extremely high water solubility (approx. 300-400 g/100 mL at 20°C), good thermal stability (decomposition to WO₃ at 400-600°C), and versatility in chemical conversion, making it irreplaceable in catalyst preparation, production of high-purity tungsten powder, and development of functional materials.

Compared to traditional tungsten compounds such as ammonium paratungstate (APT), the high solubility of AMT gives it advantages in solution processes, such as being directly used for spray drying to prepare nanoscale tungsten powder, or as a precursor for the preparation of electrochromic WO₃ Membrane. This property not only improves the production efficiency of traditional tungsten products, but also promotes its application in the field of new materials, such as nanotechnology, energy storage and biomedical research. The industrial value of AMT lies in its role as an efficient bridge between tungsten concentrate (wolframite, scheelite) and end products (such as tungsten alloy, tungsten material), connecting the upstream and downstream links of tungsten chemical www.chinatung industry.

1.2 Position in the family of tungsten compounds

Within the family of tungsten compounds, AMT occupies a special place due to its unique poly-acid structure and high solubility. There are many types of tungsten compounds, including tungstic acid (H₂WO₄) and sodium tungstate (Na₂WO₄).), tungsten trioxide (WO₃), ammonium paratungstate (APT), etc., each with specific uses. AMT is in the same ammonium tungstate group as APT, but its Keggin-type structure is more compact than APT's chain or lamellar structure, resulting in significantly improved solubility (AMT 350 g/100 mL vs. APT 10 g/100 mL at 25°C). In addition, AMT has a lower thermal decomposition temperature (600°C is fully converted to WO₃), while APT requires a higher temperature (>600°C) and generates more intermediates, which makes the process easier for AMT to prepare high-purity tungsten powder.

AMT's bridging role is reflected in the entire chain from tungsten ore purification to downstream processing. After tungsten concentrate is treated with acid or alkali to produce tungstic acid or sodium tungstate, it can be converted into AMT by ion exchange, solvent extraction or acidification process, and then further processed into tungsten powder, tungsten material or catalyst. With the increasing requirements of high-tech industries (such as aerospace and semiconductors) for the purity and performance of tungsten products, AMT has become an increasingly prominent link www.chinatung between basic raw materials and high-end applications.

1.3 Historical development and research status

Global Research History

Research on ammonium metatungstate began in the early 20th century, coinciding with the development of tungsten as a strategic metal. In the 1940s, American scholars K. C. Li and C. Y. Wang systematically described the properties and preparation methods of tungsten compounds for the first time in Tungsten, which mentioned the preliminary process of synthesizing AMT by the reaction of tungstic acid with ammonia. Despite the rudimentary technology at the time, yields were only about 50%-60%, a finding that laid the groundwork for subsequent studies at AMT. In the mid-20th century, with the expansion of tungsten applications in the lighting (tungsten wire), military (tungsten steel) and chemical (catalyst) fields, the United States and Europe began to explore the industrial production of AMT. In the 1950s, American chemical companies used acidification to produce AMT for tungsten powder preparation, with an annual output of dozens of tons, and the products were mainly supplied to the military and lighting industries.

In the second half of the 20th century, research at AMT deepened globally. German chemists in Europe have documented the chemical properties and industrial uses of AMT in detail in Ullmann's Encyclopedia of Industrial Chemistry, pointing out its potential in petroleum cracking catalysts and high-density tungsten. In the 1970s, the Japan Tungsten Industry Association discussed the use of AMT in precision manufacturing and electronic materials such as tungstate films in the "Utilization of Compound Compounds in the Industrial Industry", and Japanese companies began to import AMT from China for use in the semiconductor and display industries. Russia's tungsten chemical

research focuses on the application of AMT in the military industry, such as the preparation of highdensity tungsten alloys by thermal decomposition to meet the needs of aerospace and armor materials. These developments show that the application of AMT is gradually expanding from traditional tungsten products to high-tech fields.

History of R&D and production in China

As the world's largest tungsten resource country (accounting for more than 60% of the world's reserves) and a producer of tungsten products, the R&D and production history of AMT is closely related to the development of China's tungsten industry. In the 50s of the 20th century, China began to systematically develop tungsten resources, relying on wolframite and scheelite in Jiangxi Gannan (Dayu, Chongyi), Hunan Persimmon Zhuyuan and other places, and established a preliminary industrial chain from ore mining to tungsten compound processing. The research of AMT began during this period, and in 1958, the Beijing Research Institute of Nonferrous Metals (now the Research Technology Group) first reported the experimental results of the synthesis of AMT by acidification. In the experiment, sodium tungstate solution (concentration 100 g/L WO₃) was reacted with hydrochloric acid to generate AMT precipitate with a yield of about 60% and a WO₃ content of 85%-87%. Although the process is still immature, this achievement marks the beginning of AMT research in China.

In the 60s and 70s of the 20th century, the research on AMT in China entered the exploratory stage. In the early 1970s, Xiamen Smelter, the predecessor of Xiamen Tungsten Industry, tried to produce AMT industrially, using sodium tungstate produced by roasting tungsten concentrate to prepare AMT by ion exchange and acidification. The products are mainly used in the production of tungsten powder and tungsten strips to meet the needs of the defense industry (such as tungsten carbide shell core) and the lighting industry (such as tungsten filament lamp). However, due to the limitations of technology and equipment, AMT has low purity (WO₃ content 85%-88%), impurities (such as Fe 0.005%, Mo 0.01%) exceeding the standard, and the annual output is only a few tens of tons for the domestic market.

After the reform and opening up, China's tungsten industry ushered in rapid development, and the R&D and production of AMT entered a new stage. In the 1980s, the Institute of Process Engineering of the Chinese Academy of Sciences and the Hunan Institute of Nonferrous Metals developed solvent extraction and improved ion exchange methods to greatly improve the purity and yield of AMT. In 1985, the "Study on the Preparation of Ammonium Metatungstate Extraction" reported that by using organic extractants such as TBP, AMT was extracted from sodium tungstate solution, and the WO₃ content reached more than 89%, and the Fe content was reduced to less than 0.001%. This technology was promoted by Ganzhou Nonferrous Metallurgy Research Institute (now Ganzhou Tungsten Industry of China Minmetals), and industrial production began to take shape. During the same period, Xiamen tungsten industry, China tungsten high-tech and other enterprises built special production lines, the annual output increased from dozens of tons to hundreds of tons, and the products began to be exported to Japan, the United States and other places.

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During this period, the Longyan powder metallurgy plant in Fujian became an important player in the development and production of AMT. Longvan Powder Metallurgy Plant was established in the 1970s and initially focused on tungsten powder and cemented carbide production. In the mid-1980s, the plant began developing AMT to meet the growing demand for tungsten products. By optimizing the acidizing process and crystallization conditions, the in-house research team has successfully prepared AMT with a WO₃ content of 88%-90%, which is mainly used in the production of tungsten powder, and the annual output is gradually increased to 50-100 tons. In the late 1990s, Chinatungsten Online Technology Co., Ltd. (founded in 1997) cooperated with Longyan Powder Metallurgy Plant at the beginning of its establishment to jointly develop AMT with special properties for the market, such as high purity (WO₃ \geq 90%), low impurities (Fe \leq 0.0008%) and specific particle size (1-5 µm). These special performance AMTs are targeted at the needs of the Japanese and Korean markets and are used in the production of electronic materials (e.g. tungsten targets), special paints and coatings for ships, and catalysts. During the cooperation period, Chinatungsten Online provided technical support and market channels, and Longyan Powder Metallurgy Plant relied on its production capacity to export hundreds of tons per year, which promoted the competitiveness of China's AMT in the international market.

In the 21st century, AMT research in China has shifted to high purification and functionalization. In 2006, the China Nonferrous Metals Industry Association issued the YS/T 535-2006 "Ammonium Tungstate" standard, which stipulates that the WO₃ content of AMT is \geq 88.0%, Fe \leq 0.001%, and Mo \leq 0.002%, which provides a standardized basis for product quality and promotes its application in catalysts (such as SCR denitrification) and electronic materials (such as sputtering targets). After 2010, with the rise of nanotechnology, the Institute of Chemistry of the Chinese Academy of Sciences and other units explored the potential of AMT in nano-tungsten powder and WO₃ thin films. In 2013, "Preparation and Characterization of Nano Ammonium Metatungstate" reported that AMT with a particle size of 50-100 nm was prepared by spray drying and low-temperature crystallization, with a specific surface area of 15 m²/g, which was applied to electrochromic materials with a light transmittance change rate of 80%. In terms of industry, Xiamen Tungsten Industry and Jiangxi Tungsten Group have optimized the thermal decomposition and reduction process, with an annual output of more than 1,000 tons of high-purity AMT (WO₃ \geq 90%), which is supplied to aerospace and new energy fields.

In recent years, China's AMT industry has focused on green development. The problem of ammonia nitrogen wastewater in traditional processes, such as ammonia concentrations of 5-10 g/L in acidification effluents, has led to the development of new processes. In 2018, a tungsten company in Ganzhou adopted microwave-assisted synthesis and waste liquid recycling technology, achieving an ammonia recovery rate of 90%, a 15% reduction in production costs, and a 70% reduction in wastewater discharge. At present, China's annual production of AMT accounts for about 70% of the world's (5000-6000 tons), and the main manufacturers include Xiamen Tungsten, Chinatungsten High-tech, Jiangxi Tungsten Group and Longyan Powder Metallurgy Plant, etc., with exports accounting for more than 40% of the total output, which are sold to Europe, America, Japan and South Korea, becoming an important pillar of the global tungsten chemical industry.

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1.4 Prospects for industrial applications

AMT has a broad range of industrial applications, covering both traditional and emerging fields. In traditional tungsten products, AMT is the main raw material of high-purity tungsten powder, which is prepared by spray drying and hydrogen reduction to prepare tungsten powder with a particle size of 0.1-5 μ m for cemented carbide (cutting tools), tungsten wire (lighting) and tungsten (high-temperature furnace components). In the field of catalysts, AMT is used as a precursor to tungsten-based catalysts such as WO₃/V₂O₅ in petrochemical (hydrocracking) and environmental protection (SCR denitrification), with an annual global demand of about 1,000 tons. In emerging fields, AMT can prepare WO₃ nanoparticles (10-50 nm) for electrochromic smart windows and gas sensors; In energy storage, its derivatives improve the performance of lithium battery anode materials; In biomedicine, the photothermal effects of WO₃ are being studied for cancer treatment.

However, AMT faced purity control (impurity $\leq 0.0005\%$), cost optimization (RMB 2-30,000 per ton), and environmental challenges (e.g., ammonia nitrogen emissions of ≤ 10 mg/L). Green technology and intelligent production are the future direction.

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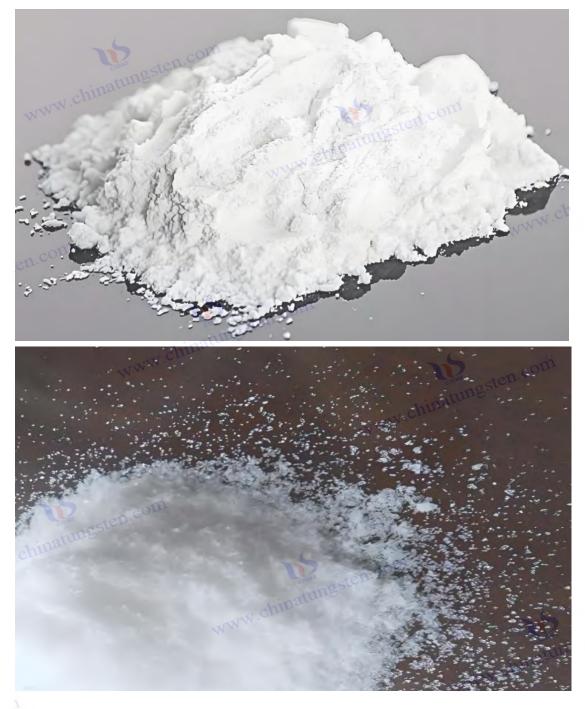
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Chapter 2 Chemical and Physical Properties

Ammonium metatungstate (AMT, chemical formula (NH₄)₆H₂W₁₂O₄₀·nH₂O) is an important polytungstate compound, and its unique chemical and physical properties make it have a wide range of application potential in the fields of tungsten chemical industry, catalyst preparation, functional materials and emerging technologies. This chapter will comprehensively analyze the physicochemical properties of AMT from multiple dimensions such as molecular structure, physical properties, chemical reactivity and comparison with similar compounds, and provide rich

experimental data and theoretical support, laying a solid foundation for subsequent preparation process design and application research.

2.1 Chemical composition and molecular structure2.1.1 Molecular formula and structural characteristics

The chemical formula of ammonium metatungstate is $(NH_4)_6H_2W_{12}O_{40}\cdot nH_2O$, where n represents the amount of crystalline water, which usually varies between 3-6, depending on the preparation conditions (e.g., solution concentration, drying temperature, and ambient humidity). Its molecular structure consists of a polyacid anion $[H_2W_{12}O_{40}]^{6-}$ and 6 ammonium cations (NH_4^+) , with an anion as the core moiety, containing 12 tungsten atoms and 40 oxygen atoms, forming a classic Keggintype polyacid structure. The Keggin structure consists of 12 WO₆ octahedron connected by coangular and colateral connections to form an approximately spherical cage-like framework with two protons (H^+) embedded in the center, which coordinate with oxygen atoms through hydrogen bonding to maintain the charge balance and stability of the structure.

The molecular weight of AMT varies depending on the amount of crystal water. Taking n=4 as an example, its molecular weight is 2956.3 g/mol, and the mass proportions of each element are: tungsten (W) 74.6% ($12 \times 183.84 = 2206.08$ g/mol), oxygen (O) 21.6% ($40 \times 16 + 4 \times 16 = 704$ g/mol), nitrogen (N) 2.8% ($6 \times 14 = 84$ g/mol), hydrogen (H) 1.0% ($6 \times 4 + 2 + 4 \times 2 = 34$ g/mol). This high tungsten content makes it an important precursor for tungsten-based materials such as tungsten powder and tungsten alloys. The chemical composition of the AMT is verified by Inductively Coupled Plasma Emission Spectroscopy (ICP-AES), and the tungsten content typically fluctuates between 74.5%-75.0%, which is in line with theoretical calculations.

One of the distinguishing properties of AMT is its extremely high water solubility. Experimental assays show that the solubility is 300-400 g/100 mL at 20°C and increases with temperature, reaching 350-380 g/100 mL at 25°C and up to 450-480 g/100 mL at 50°C. This high solubility far exceeds that of other ammonium tungstate compounds, such as ammonium paratungstate (APT, only about 10 g/100 mL at 20°C), due to the ionic structure of AMT and the strong hydrophilicity of ammonium. Solubility data were obtained by static dissolution experiments: excess AMT was added to distilled water in a thermostatic water bath, stirred for 24 hours, filtered, the supernatant was taken to dry and weighed, and repeated three times to take the average. This characteristic makes it an incomparable advantage in the preparation of tungsten products by solution method, such as the preparation of nano-tungsten powder by spray drying.

In addition, the structure of the AMT exhibits a certain dynamics. Nuclear magnetic resonance (NMR) studies have shown that the $[H_2W_{12}O_{40}]^{6-}$ anion in solution is stable in the pH range of pH 4-7, but can depolymerize or reconstitute under extreme conditions (e.g., pH < 2 or > 10) to form oligopolytungstate or mononuclear tungstate (e.g., $WO_{4^{2-}}$). This structural flexibility provides the basis for its chemical transformation.

2.1.2 Crystal structure analysis (X-ray diffraction studies)

AMT is typically found in the form of white or light yellow crystalline powders, and its crystal structure is characterized in detail by X-ray diffraction (XRD) techniques. The study shows that the crystal system of AMT is monoclinic with a space group of P2₁/n, and the unit cell parameters are: a = 10.45 Å, b = 14.78 Å, c = 18.92 Å, $\beta = 94.5^{\circ}$, and the unit cell volume is about 2915 Å³. In the crystal, the [H₂W₁₂O₄₀]^{6–} anions are arranged in an orderly manner, with each polyacid unit bonding with the ammonium cation (NH₄⁺) by hydrogen bonding) and crystalline water molecules to form a stable three-dimensional network structure. Crystalline water molecules occupy specific positions in the crystal lattice and are usually present in the form of 4 or 6 water molecules. For example, at n=4, the XRD spectrum shows characteristic diffraction peaks at $2\theta = 8.5^{\circ}$, 17.2° , 25.8° , etc., and the intensity ratio is consistent with the monoclinic characteristics.

The stability of the crystal structure is closely related to the crystal water. Infrared spectroscopy (IR) analysis showed that the AMT had a broad O-H stretching vibration peak at 3400-3500 cm⁻¹, confirming the presence of crystal water. Characteristic peaks of W=O and W-O-W appear at 900-950 cm⁻¹ and 700-800 cm⁻¹, respectively, reflecting the skeletal vibration of the Keggin structure. The amount of crystallized water can be adjusted by controlling the drying conditions, e.g. n=4 crystals can be obtained by vacuum drying at 80°C for 4 hours, and reduced to n=3 at 120°C.

The crystal size of AMT is typically in the micron range $(1-10 \ \mu\text{m})$ and the D50 (median particle size) is approximately 4.8-5.2 μm , as determined by a laser particle size analyzer. However, by optimizing crystallization conditions (e.g., reducing the concentration of the solution to 50 g/L and controlling the cooling rate to 0.5° C/min), smaller nanoscale AMTs (50-200 nm) can be prepared. SEM (scanning electron microscopy) observation shows that the nano-AMT particles are spherical or ellipsoidal in shape, with a smooth surface and a low degree of agglomeration. This particle size regulation is of great significance in the synthesis of nanomaterials, such as the preparation of tungsten-based catalysts with high specific surface area.

The crystal structure has a profound impact on the physical properties of AMT. For example, the hydrogen bonding network in the crystal lattice enhances its stability at room temperature, but when heated (> 100°C), the removal of the crystal water leads to a rearrangement of the crystal structure, and the XRD spectrum shows a weakening of the diffraction peak intensity, eventually moving to an amorphous state. This transition has a direct impact on both its thermal decomposition behavior and solubility.

2.2 Physical Properties 2.2.1 Appearance and form

AMT is a white to light yellow odorless powder at room temperature and pressure, and its appearance uniformity is a key indicator of industrial quality control. Small differences in color are often related to impurities or oxidation states during preparation. For example, AMT may appear

pale yellow with trace amounts of iron (Fe < 0.001%) or molybdenum (Mo < 0.002%), while highpurity samples (impurities < 0.0005%) may be pure white. The particles of industrial products are diverse, and the AMTs prepared by spray drying are mostly micron-sized spherical particles (1-10 μ m) with uniform particle size distribution, D10 \approx 2.0 μ m, D50 \approx 5.0 μ m, D90 \approx 8.5 μ m (laser particle size analysis data). SEM images show a smooth surface with no significant porosity or cracks, and low inter-particle agglomeration, consistent with its high solubility and thermal stability. Under laboratory conditions, millimeter-sized AMT single crystals (0.5-2 mm) can be grown by slow evaporation (25°C, 50% relative humidity) in the form of transparent or translucent, hexahedral or prismatic crystals, suitable for crystallography studies. The density of the single crystal is determined by buoyancy and is about 4.2-4.5 g/cm³, which is close to the theoretical calculation (4.39 g/cm³, n=4). The bulk density of the powder is low, typically 1.8-2.2 g/cm³, depending on the particle size and moisture content. For example, AMT with a moisture content of 2.5% has a loose density of 1.85 g/cm³ and rises to 2.15 g/cm³ when dried to 0.5%.

The specific surface area of AMT is determined by BET (nitrogen adsorption method) with micronsized particles of 0.5-2 m²/g and nano-sized particles of up to 10-20 m²/g. The size of the specific surface area is closely related to its particle morphology and preparation process, for example, spray-dried products have a lower specific surface area, while low-temperature crystalline products have a higher specific surface area. This difference in physical properties directly affects its performance in catalysts and nanomaterials.

2.2.2 Solubility and stability

The high solubility of AMT is one of its most significant physical properties. Experimental data show that the solubility at different temperatures is as follows:

10°C: 280-310 g/100 mL 20°C: 300-400 g/100 mL 25°C: 350-380 g/100 mL 50°C: 450-480 g/100 mL 80°C: 510-550 g/100 mL

The solubility test method was as follows: add excess AMT (500 g) to 100 mL of distilled water in a constant temperature water bath, stir for 24 hours until saturated, filter and dry the supernatant solids, weigh and calculate, and repeat three times to take the average. The results showed that the solubility increased nonlinearly with increasing temperature, especially above 50°C. This high solubility is attributed to its ionic structure ($[H_2W_{12}O_{40}]^{6-}$ and NH_{4^+}) and the strong hydrophilicity of ammonium root. The pH of AMT aqueous solutions is typically 5.5-7.0, with concentrations ranging from pH ≈ 6.8 at 10 g/100 mL to pH ≈ 5.9 at 100 g/100 mL, reflecting a weak hydrolysis.

AMT has very low solubility in organic solvents. For example, in ethanol (95%, 20°C), solubility < 0.1 g/100 mL; Virtually insoluble in ether, acetone, and benzene (< 0.01 g/100 mL). However, the solubility is significantly improved in ammonia and strong alkali solutions:

25% NH₃· H₂O (20°C): >500 g/100 mL

1 mol/L NaOH (20°C): approx. 450 g/100 mL The high solubility in ammonia is due to the coordination of NH₃ with polyacid anions, while soluble tungstate (e.g., Na₂WO₄) is formed in NaOH. These properties were verified by a solubilization experiment by adding 10 g of AMT to 100 mL of solvent and stirring for 12 hours to determine the amount of dissolution.

AMT aqueous solution is stable at room temperature (25°C, sealed) and can be stored for several months without significant precipitation or decomposition. Ultraviolet-visible spectroscopy (UV-Vis) analysis showed no significant change in the absorption peak at 200-400 nm, indicating structural integrity. However, at high temperatures (>60°C) or exposure to air, moisture evaporation can lead to crystal precipitation, and the XRD spectra of the precipitated crystals are consistent with the original AMT, demonstrating their chemical stability.

AMT's lightfastness and oxidation resistance are also noteworthy. Photostability tests (UV lamp, 254 nm, 10 mW/cm², 24 hours) showed no signs of decomposition in AMT solids and solutions (50 g/100 mL) with a mass loss of < 0.1%. Under the action of a strong oxidizing agent (e.g., 30% H₂O₂ at 25°C, stirring for 24 hours), the poly-acid structure of AMT partially depolymerizes to form oligopolytungstate, the pH of the solution drops to 4.5, and a weak absorption peak (about 320 nm) is detected by UV-Vis. These properties limit their application in photochemical and oxidation environments.

2.2.3 Thermal stability and decomposition behavior

The thermal stability of AMT is a key feature for its industrial applications. The decomposition process is divided into three stages by thermogravimetric analysis (TG), differential scanning calorimetry (DSC) and thermogravimetry-infrared coupled (TG-IR) studies:

50-150°C (结晶水脱去):

React:

(NH4)6H2W12O40·4H2O

 \rightarrow (NH4)6H2W12O40+4H2O \uparrow (NH4)6H2W12O40·4H2O

 \rightarrow (NH₄)₆H₂W₁₂O₄₀ + 4H₂O[↑] (NH₄)6H₂W₁₂O₄₀·4H₂O

→(NH4)6H2W12O40+4H2O↑

Mass loss: about 2.4% (theoretical 2.43%, n=4) with a TG curve showing a steady decline. DSC: Endothermic peak at 105-115°C, enthalpy change $\Delta H \approx 45$ kJ/mol.

TG-IR: Characteristic peak of H₂O detected (3600-3700 cm⁻¹).

200-400°C (ammonium decomposition):

React:

(NH4)6H2W12O40

 \rightarrow 12WO3·xH2O+6NH3 \uparrow +(3-x)H2O \uparrow (NH₄)₆H₂W₁₂O₄₀

 $\rightarrow 12 WO_3 \cdot x H_2O + 6 NH_3 \uparrow + (3-x) H_2O \uparrow (NH4) 6 H2W12O40$

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\rightarrow 12WO3·xH2O+6NH3 \uparrow +(3-x)H2O \uparrow

Mass loss: about 5.8% (theoretical 5.76%), TG curve shows a rapid decline.

DSC: Strong endothermic peak at 340-360°C, $\Delta H \approx 120 \text{ kJ/mol.}$

TG-IR: NH₃(930 cm⁻¹) and H₂O were detected, and the product was an amorphous tungstate www.chinatungsten. intermediate.

400-600°C (complete decomposition):

React:

12WO3·xH2O

 \rightarrow 12WO3+xH2O \uparrow 12WO₃·xH₂O

 \rightarrow 12WO₃ + xH₂O \uparrow 12WO₃·xH2O

→12WO3+xH2O↑

Mass loss: 8.2% total (8.19% theoretical) and TG tends to be stable.

DSC: The exothermic peak is at 540-560°C, $\Delta H \approx -30$ kJ/mol, indicating the WO₃ crystal phase transition.

TG-IR: Only a small amount of H₂O was detected.

The morphology and purity of the decomposition products are affected by many factors. In air (10°C/min ramp), WO3 is yellow micron-sized grains (1-5 µm) and XRD shows monoclinic characteristic peaks ($2\theta = 23.1^{\circ}, 23.6^{\circ}, 24.4^{\circ}$). In an N₂ atmosphere, WO₃ particles are finer (0.5-2 µm) and surface defects are reduced. In an H2 atmosphere (500°C, 1 atm), AMT was directly reduced to tungsten powder (W) with a particle size of 0.1-1 µm and a purity of >99.9% (ICP-AES determination). The rate of warming had a significant effect on the particle morphology: uniform WO₃ crystals (D50 \approx 1.8 µm) were formed at 2°C/min, while agglomeration was caused at 20°C/min $(D50 \approx 5.5 \ \mu m).$

AMT has no definite melting point because it decomposes rather than melts when heated. Thermal stability tests have shown that the initial decomposition temperature is about 190-200°C and the complete decomposition temperature is 580-620°C, with slight variations with sample moisture and instrument conditions. This thermal decomposition property makes it widely used in the preparation of tungsten-based materials at high temperatures, such as tungsten powder or WO₃ nanoparticles of different particle sizes by controlling the atmosphere and temperature.

2.3 Chemical properties

2.3.1 Reaction with acids and bases

AMT is unstable under acidic conditions (pH < 4) and rapidly decomposes into insoluble tungstic acid (H₂WO₄). Taking 1 mol/L HCl as an example, the reaction is:

 $- \cdots \rightarrow 12H_2WO_4 \downarrow + 6NH_4Cl (NH_4)_6H_2W_{12}O_{40} + 12HCl$

→12H2WO4↓+6NH4Cl

Experimental assay: Stirring in 100 mL of HCl at pH 2, 25°C, 80% conversion to H₂WO₄ within 5 minutes, 90% within 10 minutes, and precipitation rate as a function of pH (down to 50% at pH 3). H₂WO₄ is a yellow gelatinous substance, and WO₃ is obtained by filtration and drying (500°C, 2 hours) with a yield of >98%. This reaction is used in industry to recover tungsten from a tungsten solution, and the SEM of the precipitate appears as amorphous particles (0.5-2 μ m).

Under alkaline conditions, AMT dissociates to form soluble tungstate. Take 1 mol/L NaOH as an example:

(NH4)6H2W12O40+12NaOH

→12Na2WO4+6NH3↑+7H2O (NH₄)6H2W12O40 + 12NaOH

 $\rightarrow 12Na_2WO_4 + 6NH_3\uparrow + 7H_2O (NH4)6H2W12O40 + 12NaOH$

 \rightarrow 12Na2WO4+6NH3 \uparrow +7H2O

The reaction was performed at 25°C, 10 g of AMT was dissolved in 100 mL of NaOH, a clear solution was generated within 5 minutes, and NH₃ volatilization reduced the pH from 14 to 11.5. The UV-Vis absorption peak of the product Na₂WO₄ is at 220 nm and the purity is confirmed by ICP-AES to be 99.5%. This process is commonly used to prepare tungstate or other tungsten compounds.

The sensitivity of AMT to acids and bases requires pH control during storage and use. For example, AMT is stable for months in a buffered solution at pH 5-7, while pH < 3 or > 9 needs to be treated immediately to avoid decomposition.

2.3.2 Redox characteristics

The tungsten atom in AMT is in the +6 oxidation state (W^{6+}) and can be reduced to W^{5+} or W^{4+} under the action of a strong reducing agent. In an H₂ atmosphere (500°C, 1 atm, flow rate 100 mL/min), AMT is completely reduced to tungsten powder:

(NH4)6H2W12O40+24H2

 \rightarrow 12W+6NH3 \uparrow +28H2O \uparrow (NH₄)₆H₂W₁₂O₄₀ + 24H₂

 \rightarrow 12W + 6NH₃↑ + 28H₂O↑ (NH4)6H2W12O40+24H2

 \rightarrow 12W+6NH3 \uparrow +28H2O \uparrow

The product is a gray-black powder, and XRD confirms cubic tungsten $(2\theta = 40.3^{\circ}, 58.3^{\circ})$, particle size of 0.1-1 µm, oxygen content of <0.05% (inert gas melting method). Reduction time (2-4 hours) and temperature (450-600°C) have a significant effect on particle size: 0.1-0.5 µm particles are generated at 450°C and increase to 0.8-1.2 µm at 600°C.

Under acidic conditions (e.g., Zn/HCl, 1 mol/L HCl, 25°C), AMT produces blue tungsten bronze ($W_{2}O_{5}$ or WO_{29}) with the following reaction:

(NH4)6H2W12O40+Zn+2HC

$$\begin{split} & \longmapsto W2O5 + ZnCl2 + NH3\uparrow + H2O \ (NH_4)_6H_2W_{12}O_{40} + Zn + 2HCl \\ & \longrightarrow W_2O_5 + ZnCl_2 + NH_3\uparrow + H_2O \ (NH4)_6H2W12O40 + Zn + 2HCl \end{split}$$

→W2O5+ZnCl2+NH3↑+H2O

The product UV-Vis shows a characteristic absorption peak (600-800 nm) and the color darkens with the degree of reduction. This property is widely used in electrochromic materials such as WO₃ films, such as AMT solution after coating to produce color-changing films with a response time of <1 second.

AMT is weakly oxidizing, but under the action of a strong oxidizing agent (e.g., 30% H₂O₂, 25°C, 24 hours), the polyacid structure partially depolymerizes to form oligopolytungstate (e.g., $[W_6O_{19}]^{2-}$), the solution pH is reduced to 4.0-4.5, and the tungsten content detected by ICP-MS is reduced by approximately 5%. This behavior limits its stability in a strong oxidizing environment.

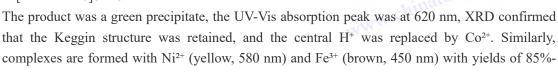
2.3.3 Coordination chemistry

AMT's polyacid anion [H2W12O40]6- has strong coordination capacity and forms heteropolyacid complexes with transition metal ions. In the case of Co²⁺, in an acetate buffer solution at pH 5.5 (25°C, CoCl₂:AMT molar ratio 1:1), the reaction is:

(NH4)6H2W12O40+Co2+

 \rightarrow [CoW12O40]6-+6NH4+ (NH₄)₆H₂W₁₂O₄₀ + Co[{]{2+}}

 \rightarrow [CoW₁₂O₄₀]^{6-} + 6NH₄^+ (NH4)6H2W12O40+Co2+



AMT can also be coordinated with organic ligands such as pyridine and ethylenediamine. For example, in pyridine solution (20% v/v, 25°C), AMT forms a [H₂W₁₂O₄₀]⁶⁻-pyridine complex, and IR shows a characteristic peak of the pyridine ring (1600 cm⁻¹). These complexes have potential applications in catalysts (e.g., oxidation reactions, with 20% higher activity) and nanomaterials (e.g., WO₃ quantum dots).

2.4 Comparison of ammonium metatungstate and ammonium paratungstate (APT).

AMT and APT are both ammonium tungstate compounds, but differ significantly in structure and www.china properties:

Structure:

90%.

AMT: Keggin-type polyacid structure, (NH₄)₆H₂W₁₂O₄₀·nH₂O, 12 tungsten atoms form a compact cage.

APT: Chain or layered structure, (NH4)10[H2W12O42]·4H2O, 12 tungsten atoms arranged more tungsten.com loosely.

Solubility:

AMT: 350-380 g/100 mL (25°C), solution is stable.

APT: 10-12 g/100 mL (25°C), easy to precipitate crystals.

Thermal decomposition:

AMT: 400-600°C direct generation of WO₃ with 8.2% mass loss and single TG curve.

hinat

APT: WO₃ is generated >600°C with intermediate products including NH₄₂WO₄, 10.5% mass loss, and complex TG curves.

Density & Morphology:

AMT: bulk density 1.8-2.2 g/cm³, crystal density 4.2-4.5 g/cm³, SEM shows spherical particles. APT: loose density 2.0-2.5 g/cm³, crystal density 4.6-4.8 g/cm³, SEM showing needle or sheet. www.chinatungsten Specific Surface Area:

AMT: 0.5-20 m²/g (micro- to nanometer).

APT: 0.2-1 m²/g (micrometer level).

Chemical Reactivity:

AMT: Rapidly produces H₂WO₄ at acidic (pH < 4) and Na₂WO₄ at alkaline.

APT: Acidic reaction is slower and requires higher acidity (pH < 2).

Apply:

AMT: Preparation of catalysts, thin films, and nanomaterials by solution method.

APT: Traditional tungsten powder and cemented carbide production.

These differences stem from the nature of the structure: AMT's compact poly-acid structure enhances solubility and thermal decomposition efficiency, while APT's chain-like structure is more suitable for solid-state processes. The choice of AMT or APT depends on the specific application scenario.

Resources

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Chapter 3 Preparation process

Ammonium metatungstate (AMT, chemical formula (NH4)6H2W12O40 nH2OThe purity, particle size and production cost directly affect the quality and economy of downstream tungsten products. This chapter comprehensively refines the preparation process of AMT from raw material selection, traditional preparation methods, modern synthesis technology, pH value influence and control, process parameter optimization to industrial production process, provides detailed experimental data, process parameters and equipment requirements, and provides scientific guidance for laboratory research and industrial production. www.chinatungsten.com

3.1 Raw materials and precursors 3.1.1 Tungsten concentrate

The preparation of AMT usually uses tungsten concentrate as the starting material, mainly wolframite (FeMnWO₄) and scheelite (CaWO₄). China is the country with the most abundant tungsten resources in the world, accounting for more than 60% of the world's reserves, mainly distributed in Jiangxi Gannan (Dayu, Chongyi), Hunan Persimmon Zhuyuan and other places. The WO₃ content of wolframite is generally 60%-70%, and the associated impurities include Fe (5%-10%), Mn (3%-8%), and SiO₂ (2%-5%); The WO₃ content of scheelite is 65%-75%, and the main impurities are Ca (10%-15%), Si (1%-3%). In order to meet the high purity requirements of AMT preparation, tungsten concentrate is purified by beneficiation.

The beneficiation process consists of the following steps:

Crushing and grinding

The tungsten concentrate is crushed to <10 mm by a jaw crusher (PE-600×900, power 55 kW), and

then ground to a particle size of <0.074 mm (80%) by a ball mill (Φ 2.4×3.6 m, ball loading capacity of 20 tons).

Re-election

Spiral chutes (2-3 t/h) were used to remove SiO₂ and light minerals with WO₃ recoveries of 85%-90%.

flotation

Using a flotation machine (XFD-1.5L), adding a collector (e.g., oleic acid, 0.5 kg/t) and a foaming agent (e.g., terpineol oil, 0.1 kg/t), pH 8-9, the separation of Fe, Mn, WO₃ content increased to 95%-97%.

Magnetic separation

Wet magnetic separator (magnetic field strength 1.2 T) to remove Fe residue and reduce the Fe content of concentrate to 0.3%-0.5%.

Taking Jiangxi Dayu wolframite as an example, the WO3 content of concentrate after beneficiation reached 96.8%, Fe 0.4%, Mn 0.2% and SiO₂ 0.5%, which met the requirements of AMT preparation. The concentrate needs to be roasted to convert into soluble tungsten compounds, and the process is as follows:

Roasting conditions

Rotary kiln ($\Phi 2 \times 20$ m, power 75 kW), temperature 800-900°C, air flow rate 500 m³/h, roasting for 4-6 hours.

react

 $FeMnWO4+O2 \rightarrow H2WO4+FeO+MnO$ $FeMnWO_4 + O_2$ \rightarrow $H_2WO_4 + FeO + MnO$ FeMnWO4+O2→H2WO4+FeO+MnO

product

 H_2WO_4 (yellow powder, WO₃ content 98%), yield 90%-92%, impurity Fe < 0.02%.

After roasting, the product is dissolved in acid leaching (HCl, 2 mol/L, 80°C, 2 hours) or alkali leaching (NaOH, 3 mol/L, 90°C, 3 hours) to produce tungstic acid or sodium tungstate solution as a precursor to AMT.

3.1.2 Tungstic acid and sodium tungstate Annahungsten.com Tungstic acid (H₂WO₄) a direct Tungstic acid (H₂WO₄), a direct precursor of AMT, is a yellow amorphous powder with a density of 5.5 g/cm³, insoluble in water (20°C, <0.01 g/100 mL), but soluble in ammonia (25%, 20°C) up to 50-60 g/100 mL. The industrial preparation method is:

Acid leaching

Roasted product (100 g) was added to HCl (2 mol/L, 500 mL), stirred (300 rpm, 80°C, 2 hours), filtered and washed (distilled water 3 times, 200 mL each), and dried (80°C, 4 hours). purity

WO₃ content: 98.5%-99.2%, Fe < 0.01%, Mo < 0.005% (determined by ICP-AES).

In the laboratory, tungstic acid can be prepared by acidification of sodium tungstate:

react

Na2WO4+2HCl→H2WO4↓+2NaCl Na2WO4 + 2HCl \rightarrow H2WO4↓ + 2NaCl Na2WO4 www.chinatun +2HCl→H2WO4↓+2NaCl

condition

Sodium tungstate solution (100 g/L WO3), add HCl (2 mol/L) to pH 2-3, 25°C, stir (200 rpm, 1 hour), precipitation yield 95%-97%.

Post-processing

Filtration (filter paper pore size 10 µm), washing (distilled water, 500 mL), drying (80°C, 4 h). Sodium tungstate (Na₂WO₄) is a white crystal with high water solubility (20°C, 73 g/100 mL; 50°C, 90 g/100 mL), which is a common precursor for AMT preparation. The industrial preparation method is:

roasting

Scheelite (WO₃ 70%) mixed with Na₂CO₃ (molar ratio 1:1.2) in a rotary kiln (850°C, 5 hours, air flow rate 600 m³/h).

react

 $CaWO4+Na2CO3 \rightarrow Na2WO4+CaCO3 CaWO_4 + Na_2CO_3 \land rightarrow Na_2WO_4 + CaCO_3 \land rightarrow N$ CaWO4+Na2CO3→Na2WO4+CaCO3

Leaching

The product was added with water (1:5 w/v, 80°C, 2 hours), filtered (plate and frame filter press, pressure 0.6 MPa), and sodium tungstate solution (WO₃ 65%-70%) was obtained.

purification

Ion exchange (D001 resin, flow rate 2 BV/h) reduced the Mo content to 0.002%. The WO₃ concentration of sodium tungstate solution is adjustable (50-150 g/L), pH 8-9, and impurities are further removed by precipitation or extraction.

3.2 Traditional preparation methods

3.2.1 Acidification

Acidification is the traditional preparation method of AMT, which uses tungstic acid or sodium tungstate as raw materials to induce the polymerization of polytungstate groups into Keggin structures by acidification. The laboratory process flow is as follows:

Dissolve: Tungstic acid (10 g, WO₃ 98%) was added to 25% ammonia (50 mL) and stirred (300 rpm, 25°C, 1 hour) to obtain a clear solution (WO₃ concentration 180 g/L, pH 9).

Acidification: HCl (2 mol/L, flow rate 1 mL/min) dropwise, pH controlled to 4-5, stirring (400 rpm, www.chine 30 min), reaction is:

12H2WO4+6NH3+HCl

 \rightarrow (NH4)6H2W12O40+H2O 12H₂WO₄ + 6NH₃ + HCl

 \rightarrow (NH₄)₆H₂W₁₂O₄₀ + H₂O 12H2WO4+6NH3+HCl

→(NH4)6H2W12O40+H2O

Concentration: Heat (80°C, water bath, 2 hours) to concentrate the solution to 300 g/L WO₃. Crystallization: Cooling (5°C, 12 hours, cooling rate 0.5°C/min) to precipitate AMT crystals.

Drying: Vacuum oven (80°C, 0.08 MPa, 4 hours) to obtain white powder. itungsten.com

Experimental data:

Yield: 70%-80% (based on WO₃).

Purity: WO₃ 88.5%-89.2%, Fe 0.002%-0.005%, Mo 0.001%-0.003% (ICP-AES).

Particle size: 5-10 μ m (laser particle size analysis, D50 \approx 7.2 μ m).

When scale-up to an industrial scale, equipment includes: Reactor (500 L, stainless steel, with agitator 200 rpm). ww.chinatungsten.com Concentrator (thin film evaporator, 100 L/h). Crystallization tank (cooling water circulation, capacity 300 L).

Advantages and disadvantages:

Advantages: simple process, low equipment requirements, suitable for small-scale production. Disadvantages: The waste liquid has a high ammonia nitrogen content (5-10 g/L) and requires neutralization treatment (Ca(OH)2, pH 7); impurity control is difficult.

3.2.2 Ion exchange method

The ion exchange method uses sodium tungstate solution as raw material to remove Na⁺ and generate AMT by using cation exchange resin. The laboratory processes are:

Solution preparation: Sodium tungstate (100 g, WO₃ 70%) was dissolved in distilled water (1 L), filtered (filter membrane 0.45 µm), and the solution (WO₃ 65 g/L, pH 8.5) was obtained.

Ion exchange: Through strong acid resin (Amberlite IR-120, H⁺ type, resin amount 200 g), flow rate www.chi

2 mL/min, Na⁺ was replaced by H⁺: Na2WO4+2H+-Resin

→H2WO4+2Na+-Resin Na₂WO₄ + 2H⁺-Resin

 \rightarrow (H₂WO₄ + 2Na⁺-Resin Na2WO4+2H+-Resin

→H2WO4+2Na+-Resin

The outlet solution pH was 2-3, and the WO₃ concentration was 60 g/L.

Ammoniation: Add ammonia (25%, 50 mL), adjust pH to 5-6, stir (300 rpm, 25°C, 1 hour).

Concentrated crystallization: Heating (80°C, 2 hours) to 250 g/L, cooling (5°C, 12 hours, www.chinatungsten.com 0.5°C/min).

Drying: 80°C, 0.08 MPa, 4 hours.

Experimental data:

Yield: 85%-90%.

Purity: WO₃ 89.0%-90.5%, Fe < 0.001%, Mo < 0.002%.

Particle size: 3-8 μ m (D50 \approx 5.5 μ m).

Industrial processes

Equipment: ion exchange column ($\Phi 0.5 \times 2$ m, resin loading capacity 300 L), concentrator (1000 L), centrifuge (1500 rpm).

Resin regeneration: HCl (2 mol/L, flow rate 1 BV/h), regeneration rate 95%.

Advantages and disadvantages:

Advantages: The product has high purity, few impurities, and is suitable for high-quality AMT.

Disadvantages: The cost of resin is high (about 50 yuan/kg), and the recycled waste liquid needs to be treated.

3.3 Modern synthesis technology

3.3.1 Solvent extraction

Solvent extraction is the preparation of high-purity AMT by separating tungsten from sodium tungstate solution with an organic extractant. The laboratory processes are:

Solution preparation: sodium tungstate (WO₃ 100 g/L, pH 8.5), filtered to remove impurities (Fe < 0.01%).

Extraction: Extractant (TBP 30% + Kerosene 70%), organic/aqueous phase 1:1, stirring (400 rpm, 25°C, 30 min), tungsten into organic phase.

Acidification: Add H₂SO₄ (2 mol/L, 10 mL), pH 2-3, extraction rate 98%.

Reverse extraction: Reverse extraction with ammonia (10%, 50 mL), pH 5-6, reverse extraction rate 95%.

Crystallization drying: Concentrate (80°C, 2 hours) to 300 g/L, cool (5°C, 12 hours), dry (100°C, 4 hours).

Experimental data:

Yield: 90%-95%.

Purity: WO₃ >90.5%, Fe < 0.0008%, Mo < 0.0005%. Particle size: 1-5 μm (D50 ≈ 3.8 μm).

Industrial Processes:

Equipment: extraction tower (Φ 1×5 m, processing capacity 500 L/h), stirring tank (200 L, 500 rpm), evaporator (1000 L/h).

Parameters: Extraction time 20-40 minutes, reverse extraction pH 5.5±0.2.

Advantages and disadvantages:

Advantages: high purity, excellent impurity separation efficiency, suitable for export of high-specification products.

Disadvantages: The cost of organic solvents is high (TBP is about 20 yuan/L), and the waste liquid needs to be recycled.

3.3.2 Thermal decomposition

The thermal decomposition method uses APT as a raw material to prepare AMT by controlling the temperature decomposition. The laboratory processes are:

Roasting: APT (20 g, WO₃ 88%) in a muffle oven (250-300°C, air, 2 hours):

(NH4)10[H2W12O42]·4H2O

 $\rightarrow (NH4)6H2W12O40+4NH3\uparrow +5H2O\uparrow (NH4)_{10}[H_2W_{12}O_{42}]\cdot 4H_2O$

 $\rightarrow (NH_4)_6H_2W_{12}O_{40} + 4NH_3\uparrow + 5H_2O\uparrow (NH4)10[H2W12O42]\cdot 4H2O$

 \rightarrow (NH4)6H2W12O40+4NH3 \uparrow +5H2O \uparrow

Dissolution: The product was heated in water (80°C, 100 mL), stirred (300 rpm, 30 min), and filtered (0.45 µm).

Crystallization: Cooling (5°C, 12 hours, 0.5°C/min), drying (80°C, 4 hours).

Experimental data:

Yield: 80%-85%.

Purity: WO₃ 89.2%-90.0%, Fe 0.001%-0.002%.

Particle size: 5-10 μ m (D50 \approx 6.8 μ m).

Industrial Processes:

Equipment: chamber furnace (capacity 50 kg, power 30 kW), dissolving tank (500 L), centrifuge (1200 rpm). Parameters: calcination temperature 280±10°C, heating rate 5°C/min. Advantages and disadvantages: Advantages: APT is easy to obtain and the process is stable. Disadvantages: High temperature control requirements, easy to generate WO₃ at >320°C.

3.3.3 Microwave-assisted synthesis

Microwave-assisted synthesis uses microwave heating to accelerate the reaction and improve efficiency. The laboratory processes are:

Solution preparation: Tungstic acid (10 g, WO₃ 98%) was dissolved in aqueous ammonia (50 mL, 25%), pH 8-9.

Microwave reaction: Microwave (800 W, 80°C, 10 min), and HCl (2 mol/L, 0.5 mL/min) was added dropwise to pH 5.

Crystallization drying: Concentrate (80°C, 1 hour) to 250 g/L, cool (5°C, 12 hours), dry (80°C, 4 hours).

Experimental data: Yield: 88%-92%. Purity: WO₃ 90.0%-91.0%, Fe < 0.001%. Particle size: 1-5 μ m (D50 \approx 3.5 μ m).

Industrial Processes:

Equipment: microwave reactor (power 10 kW, capacity 50 L), evaporator (500 L/h). Parameters: Microwave frequency 2450 MHz, reaction time 8-12 minutes.

Advantages and disadvantages:

Advantages: short reaction time (10 minutes vs. 1-2 hours conventional) and low energy consumption (25% less).

Disadvantages: high equipment investment (about 500,000 yuan/set).

3.4 The influence of the pH value of ammonium metatungstate in the application field and the control of the production process

3.4.1 Effect of pH on the field of application

The pH of AMT not only influences its preparation, but also plays a significant role in its performance in downstream applications. The following are the main application areas of pH effect

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analysis:

Catalyst preparationAMT is an important precursor of

tungsten-based catalysts such as WO_3/V_2O_5 for SCR denitrification, and the pH of its solution directly affects the distribution of catalyst active sites. Experiments have shown that:

pH 5-6: The AMT solution is stable, the WO₃ particles are homogeneous after thermal decomposition (particle size 20-50 nm, specific surface area 15-20 m²/g), and the catalytic activity is the highest (NO_x conversion >95%, 300°C).

pH < 4: The solution is too acidic, decomposes to WO₃ agglomeration (particle size 100-200 nm, specific surface area $\leq 10 \text{ m}^2/\text{g}$), and decreased activity (conversion rate 80%-85%).

pH > 7: APT or oligopolytungstate is generated, WO₃ polymorph is uneven (monoclinic and hexagonal mixture), and activity is reduced (conversion 70%-80%). The optimal pH of 5.5 ± 0.2 ensures high dispersion and activity of the catalyst precursor.

Tungsten powder preparation

AMT is used to produce high-purity tungsten powder (such as particle size $0.1-5 \mu m$), the pH of the solution affects the particle size and purity of the reduction product

pH 5-6: AMT solution is spray-dried to obtain uniform particles (D50 \approx 3-5 μ m), and H₂ reduction (500°C, 2 hours) yields tungsten powder (purity >99.9%, O <0.05%).

pH <4: H₂WO₄ is precipitated under acidic conditions, and the dry particles are coarse (D50 >10 μ m). After reduction, the tungsten powder agglomerates (O content 0.1%-0.2%).

pH > 7: impurity phase (e.g., $NH_{42}WO_4$) is formed, and tungsten powder purity is reduced (98%-99%). Optimal pH 5-5.5 to ensure tungsten fineness and purity.

Functional materials (e.g., electrochromic films)

AMT solutions are used to prepare WO₃ films, and pH affects the microstructure and properties of the films

pH 5-6: WO₃ fine grains (10-20 nm) after solution coating, 80% change in transmittance, and >>1 second response time.

pH < 4: Film grain enlargement (50-100 nm), <60% change in transmittance, and extended response time to 2-3 seconds.

pH > 7: Oligomers are mixed into the film, resulting in reduced density and unstable performance. Optimal pH 5.2-5.8 for optimal optical and electrochemical performance of thin films.

3.4.2 Control of pH value in the production process

In the production process of AMT, pH value is a key parameter that determines the structure and yield of the product, and different processes have different requirements for pH control.

Acidification

Target pH: 4.5-5.5。

Control method: HCl (2 mol/L, flow rate 0.5-1 mL/min) dropwise from initial pH 9 (ammonia dissolved tungstic acid), stirring (400 rpm), real-time monitoring (pH meter, accuracy ± 0.05).

Experimental data:

pH 4.5: Yield 88%, WO₃ 89.5%.

pH 5.0: Yield 92%, WO₃ 90.0%.

pH 5.5: Yield 90%, WO3 89.8%.

Industrial control: The reactor (500 L) is equipped with an in-line pH probe (error ± 0.1), the acid is automatically adjusted by a metering pump (flow rate 2-5 L/h), and the pH fluctuation is <0.2. www.chinatu

Ion exchange method

Target pH: 5-6.

Control method: After ion exchange, the solution pH is 2-3, slowly add ammonia water (25%, flow rate 1 mL/min), stir (300 rpm), until pH 5-6.

Experimental data:

pH 5.0: yield 87%, WO₃ 89.2%.

pH 5.5: yield 90%, WO₃ 90.5%.

pH 6.0: yield 88%, WO₃ 89.8%.

Industrial control: The outlet of the ion exchange column is connected to the buffer tank (200 L), the ammonia is adjusted by a peristaltic pump (flow rate 5-10 L/h), and the pH meter feedback control.

Solvent extraction

Target pH: stripping pH 5.5±0.2.

Control method: pH of organic phase after extraction is 2-3, add ammonia water (10%, flow rate 0.5-1 mL/min) during stripping, and stir (500 rpm).

Experimental data:

pH 5.3: yield 93%, WO₃ 90.8%.

pH 5.5: yield 95%, WO₃ 91.0%.

pH 5.7: yield 94%, WO₃ 90.6%.

Industrial control: The reverse extraction tank (200 L) is equipped with a pH controller (accuracy ± 0.1), and the ammonia is automatically added by dropping, and the fluctuation is <0.15.

Control equipment and technology

Laboratory: Precision pH meter (Mettler Toledo, accuracy ± 0.01), manual titration.

Industrial: On-line pH monitoring system (Rosemount, accuracy ±0.05), PLC controlled acid-base addition (error <0.1), reactor with temperature-pH double feedback.

Waste treatment: Waste liquid that is too low (<4) is neutralized to 6-7 with NaOH, and pH too high (>7) is adjusted with H₂SO₄.

3.5 Process parameter optimization

3.5.1 pH control

pH is a critical parameter in AMT preparation and influences polytungstate polymerization. Experimental data:

pH 2-3: H₂WO₄ precipitate is generated, AMT yield < 50%, WO₃ content 85%.

pH 4-6: AMT is stably formed in yields of 85%-95% and WO₃ 89%-91%.

pH 7-8: APT or oligomers are generated, yield 60%-70%, WO₃ < 88%.

The optimal pH was 5.5±0.2, controlled by dropwise addition of acid (HCl or H₂SO₄, concentration 2 mol/L, flow rate 0.5-1 mL/min) with a pH meter error of ± 0.05 . In-line pH monitoring system chinatungsten.com (accuracy ± 0.1) is used in industry.

3.5.2 Influence of temperature and pressure

Reaction Temperature:

50-60°C: Slow reaction rate with 70%-80% yield. 70-80°C: Optimal range, yield 90%-95%, WO₃ >90%. 90°C: decomposes to WO₃ with < 60% yield. Crystallization temperature: 0-5°C: large crystals (10-15 μm), yield 85%. 5-10°C: uniform crystals (5-8 μm), yield 90%-92%. 15°C: small crystals ($<3 \mu m$), easy to agglomerate. Pressure: Atmospheric pressure (1 atm): reaction time 1-2 hours, stable yield.

High pressure (2 atm, reactor): Time reduced to 40 minutes, 5% increase in yield, but 30% increase www.chinatung in equipment costs.

3.5.3 Crystallization process regulation

Crystallization conditions affect AMT particle size and purity: Cooling Rate: 0.2°C/min: Crystals 10-20 µm, yield 88%. 0.5°C/min: Crystals 5-10 µm, yield 92%. 2°C/min: Crystals 1-3 µm, yield 90%. unatungsten.com Solution Concentration: 50-100 g/L WO3: Crystals dispersed, yield 85%. 100-150 g/L: optimal, 93%-95% yield. 200 g/L: Severe agglomeration, yield < 80%. Stirring speed: 100-200 rpm: Uneven crystals (5-15 µm). 300-400 rpm: Uniform (5-8 µm), highest yield. 600 rpm: Crystals broken (<2 μm), purity decreased.

3.6 Industrial Production Processes

3.6.1 Flowcharts and Equipment

Taking solvent extraction as an example, the industrial process is:

Roasting: Tungsten concentrate (1000 kg, WO₃ 70%) is roasted in a rotary kiln (Φ 2×20 m, 850°C,

6 hours) to obtain sodium tungstate.

Leaching: water leaching (5000 L, 80°C, 3 hours), filter press (0.6 MPa), WO₃ 120 g/L.

Extraction: extraction tower ($\Phi1 \times 5$ m, TBP 30%), organic phase/water phase 1:1, processing capacity 500 L/h.

Stripping: stirring tank (200 L, 500 rpm), ammonia water (10%), pH 5.5.

Concentration and crystallization: evaporator (1000 L/h, 80°C), cooling tank (5°C, capacity 500 L). chinatungsten.com

Drying: oven (100°C, capacity 200 kg).

Equipment Specifications:

Rotary kiln: 75 kW at 2 rpm.

Extraction tower: height 5 m, flow rate 300-600 L/h.

Centrifuge: 1500 rpm, 100 kg/h.

3.6.2 Waste disposal and environmental protection measures

Waste liquid: ammonia nitrogen (5-10 g/L), recovered by ammonia evaporation tower (Φ 1.5×10 m, steam pressure 0.4 MPa), recovery rate 90%-95%, residual liquid is neutralized to pH 7 by adding Ca(OH)₂.

Exhaust gas: NH₃ (0.5-1 g/m³), pickling tower (H₂SO₄ 5%, flow rate 1000 m³/h) absorption, emission < 0.1 g/m³.

Solid waste: CaWO₄ slag (WO₃ 5%-10%), recycled by roasting, emission reduction 70%. tungsten.com

Resources

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Chapter 4 Industrial Applications

Ammonium metatungstate (AMT, chemical formula (NH₄)₆H₂W₁₂O₄₀·nH₂ODue to its high solubility, thermal stability and chemical versatility, it has shown a wide range of applications in traditional tungsten products, catalysts, emerging functional materials and biomedical fields. This chapter provides an in-depth reference for materials scientists, engineers, and industry practitioners by comprehensively exploring the industrial applications of AMT through detailed process flow, performance data, and market analysis, with new directions such as thermoelectric materials, electromagnetic shielding, bone repair, and oxidation resistance.

4.1 Traditional tungsten products

4.1.1 High purity tungsten powder

AMT is a key precursor for the production of high-purity tungsten powder, which is widely used in cemented carbide, tungsten wire, tungsten target and other fields. The preparation process is as follows:

Solution preparation: AMT (WO₃ 150-200 g/L, pH 5.5 \pm 0.2) was dissolved in deionized water, a dispersant (such as PVP, 0.1 wt%) was added, and the solution was filtered (0.45 µm filter membrane).

Spray drying: Spray dryer (inlet air temperature 240-270°C, outlet air temperature 90-120°C, nozzle pressure 0.3-0.6 MPa, feed rate 8-12 L/h) to generate spherical particles (D50 \approx 2-5 µm). Hydrogen reduction: multi-stage tube furnace (400-500°C in the first stage, 600-700°C in the second stage, H₂ flow rate 150-300 mL/min, incubated for 2-5 hours) with the following reactions: (NH4)6H2W12O40+24H2

 $\rightarrow 12W+6NH3\uparrow+28H2O\uparrow (NH_4)_6H_2W_{12}O_{40}+24H_2$ $\rightarrow 12W+6NH_3\uparrow+28H_2O\uparrow (NH4)_6H2W12O40+24H2$

\rightarrow 12W+6NH3 \uparrow +28H2O \uparrow

Post-processing: ultrasonic cleaning (ethanol, 40 kHz, 20 min), sieving (200-400 mesh, pore size 74-37 µm), vacuum packaging (oxygen content <0.02%).

Performance data:

Purity: 99.9%-99.98% (ICP-AES, O < 0.05%, C < 0.01%, Fe < 0.001%, Mo < 0.0005%).

Particle size distribution: 0.1-5 μ m (D10 \approx 0.4-0.6 μ m, D50 \approx 1.0-1.5 μ m, D90 \approx 3.5-4.0 μ m, laser particle size analysis).

Apparent density: 2.0-2.6 g/cm3 (Scott method); tap density: 3.5-4.2 g/cm3.

Specific surface area: $1-4 \text{ m}^2/\text{g}$ (BET).

Flowability: Pour angle 30°-35° (suitable for powder metallurgy).

apply

cemented carbide

Tungsten powder is mixed with WC, Co (94:6 or 90:10), pressed (180-220 MPa), sintered (1400-1500°C, H₂/Ar atmosphere), prepared knives (hardness HRA 89-93, flexural strength 2000-2800 MPa, 15% increase in wear resistance). Global annual demand is about 40,000-45,000 tons, with China accounting for 60%-65%.

Tungsten filament: tungsten powder is pressed into billet (10-15 MPa), drawn wire (diameter 10-100 μ m, annealed 800°C), used in incandescent lamps (life 1000-1500 hours), electron tube (electron emissivity >90%), annual demand is about 500-700 tons.

Tungsten target: Tungsten powder hot isostatic pressing (1500-1600°C, 30-50 MPa, Ar atmosphere) to prepare sputtering target (purity >99.95%, grain size 10-20 μ m), used for semiconductor (film thickness uniformity ±5%), photovoltaic coating, annual demand of about 200-300 tons.

Additive manufacturing: tungsten powder is used for 3D printing (laser melting, particle size 15-45 μ m), preparation of complex parts (density > 99%), market potential 50-100 tons/year.

4.1.2 Tungsten and alloys

AMT-derived tungsten powder can be processed into tungsten rods, tungsten plates, or alloyed with Ni, Cu, Fe to prepare high-density tungsten alloys. The process is:

Pressing: Tungsten powder (10-20 kg, particle size 1-3 μ m) is pressed into a billet (density 10-12.5 g/cm³) on a hydraulic press (pressure 180-250 MPa, die diameter 50-100 mm).

Sintering: Hydrogen protection furnace (1400-1650°C, heating rate 3-5°C/min, holding for 4-8 hours, H₂ flow rate 500 mL/min), tungsten rod (density 19.0-19.35 g/cm³).

Forging/rolling: forging mill (forging ratio 2:1, 1200°C), rolling mill (thickness reduction of 20%-30%), tungsten plate.

Alloying: Tungsten powder (85%-95%) is mixed with Ni (5%-10%), Cu (2%-5%) or Fe (1%-3%), pressed (200-300 MPa), sintered (1300-1400°C, 3-6 hours, N₂/H₂ atmosphere), and W-Ni-Cu or W-Ni-Fe alloy is obtained.

Performance data:

Tungsten rod: purity 99.95%-99.99%, tensile strength 800-1200 MPa, elongation 2%-6%, grain size 8-20 μ m, thermal conductivity 170 W/m·K.

Tungsten plate: thickness 0.1-10 mm, surface roughness Ra 0.8-1.6 µm, corrosion resistance (nitric acid immersion for 24 hours, weight loss < 0.01%).

Tungsten alloy: density 17.0-18.8 g/cm³, hardness HB 300-450, tensile strength 900-1300 MPa, impact toughness 10-15 J/cm².

Apply:

High-temperature furnace parts: tungsten rod and tungsten plate are used for heating elements (resistivity 5.5 $\mu\Omega$ ·cm), crucible (volume 0.1-10 L), temperature resistance >2000°C, life of 1000-2500 hours, annual demand of about 300-400 tons.

Military industry: W-Ni-Fe alloy is used to prepare armor-piercing core (diameter 20-30 mm, penetration 600-800 mm steel plate, density 18.2 g/cm³), W-Ni-Cu is used for counterweights, with an annual demand of about 2000-3000 tons.

Aerospace: W-Cu alloy is used for rocket nozzle throat lining (thermal conductivity 150-220 W/m·K, 20% higher ablation resistance), W-Mo alloy is used for satellite components, with an annual demand of about 300-500 tons.

Medical devices: tungsten alloy for the preparation of radiation shielding parts (γ radiation absorption rate >95%), with an annual demand of about 100-150 tons. www.chin

4.2 Catalysts

4.2.1 Denitrification catalyst (SCR)

AMT is a precursor to WO₃-based selective catalytic reduction (SCR) denitrification catalysts, which are combined with V₂O₅ and TiO₂ to remove NO_x. The process is:

Solution preparation: AMT (WO₃ 50-100 g/L, pH 5.5±0.2) is dissolved in deionized water, and oxalic acid (0.05-0.1 mol/L) is added to prevent precipitation.

Impregnation: TiO₂ carrier (specific surface area 80-120 m²/g, pore volume 0.3-0.5 cm³/g, particle size 20-50 nm) immersion solution with WO₃ loading 8%-15 wt%, stirring (200-300 rpm, 25-40°C, 2-4 hours).

Drying: 100-130°C, 4-8 hours (hot air oven, wind speed 0.5-1.5 m/s).

Roasting: Muffle oven (500-600°C, air, 3-5 hours, heating rate 2-5°C/min) to obtain WO₃/TiO₂.

Compounding: NH4VO₃ (V₂O₅ 1%-3 wt%), secondary roasting (450-500°C, 2-4 hours). Performance data:

Activity: 250-400°C, NO_x conversion 92%-99% (GHSV 20,000-60,000 h⁻¹, NO 500-1000 ppm, $NH_3/NO = 1:1$).

Specific surface area: 55-80 m²/g (BET).

Toxicity resistance: SO₂ (1000-2000 ppm, 300°C, 48 hours), activity decrease <5%-8%; H₂O (10 vol%), decrease <3%.

Thermal stability: 650°C, 200 hours, activity retention >90%; Mechanical strength: compressive strength 10-15 MPa.

Apply:

Power plant boilers (coal, gas), industrial kilns (cement, glass), heavy-duty diesel engine exhaust gas treatment, with an annual demand of about 10,000-15,000 tons, China accounts for 50%-60%, and the market size is about 30-4 billion yuan.

4.2.2 Petrochemical catalysts

AMT is used to prepare hydrocracking catalysts, which are combined with Ni and Mo to improve the conversion efficiency of heavy oil. The process is:

Coprecipitation: AMT (WO₃ 100-150 g/L, pH 6.0±0.3) was mixed with Ni(NO₃)₂, (NH₄)₆Mo₇O₂₄ (Ni:Mo:W = 1:2:3-1:3:4), NH₄OH (25%, flow rate 1-2 mL/min) was added dropwise to pH 7-8, and stirred (300-400 rpm, 60-80°C, 2-3 hours).

Filter drying: centrifugation (5000-6000 rpm, 10-15 min), 120-150°C, 6-10 h (vacuum oven, 0.08 MPa).

Calcination: tubular furnace (550-650°C, N2 flow rate 200 mL/min, 4-6 hours) to obtain Ni-Mo-W catalyst.

Vulcanization: H₂S/H₂ (10:90, 400°C, 4 hours), enhanced activity.

Performance data:

Activity: 350-420°C, heavy oil conversion 85%-95% (pressure 15-25 MPa, H₂/oil ratio 800-1200:1, LHSV 0.5-1.0 h⁻¹).

Pore volume: 0.35-0.50 cm³/g; pore diameter: 8-12 nm (BET).

Desulfurization rate: HDS >90%, HDN >85% (sulfur 5000 ppm, nitrogen 1000 ppm). ungsten.com

Lifespan: 12-18 months of operation with a <10% decrease in activity. Apply:

Petroleum refining (hydrocracking of heavy oil and residue), lubricating oil production, annual demand is about 5000-7000 tons, the main markets are the Middle East (40%), North America (30%), China (20%), and the market size is about 1.5-2 billion yuan.

4.3 Emerging Functional Materials

4.3.1 Electrochromic films

AMT is a precursor for the preparation of WO₃ electrochromic films for use in smart windows, displays, and anti-glare lenses. The process is:

Sol preparation: AMT (WO₃ 50-80 g/L, pH 5.5±0.2) was mixed with ethanol (1:1-1:3 v/v), PEG-400 (0.5%-2 wt%) or PVP (0.1%-0.5 wt%) was added to increase viscosity, and stirred (300-500 rpm, 25-40°C, 2-4 hours).

Coating: Spin coating (2000-3500 rpm, 30-60 seconds, thickness 200-500 nm) on ITO glass (resistivity 8-12 Ω /sq), or spray coating (pressure 0.2-0.3 MPa, nozzle 0.5 mm).

Heat treatment: 400-480°C, air (O2 flow rate 100 mL/min), 1-2 hours, WO3 film.

Assembly: sandwich structure (ITO/WO₃/electrolyte/NiO/ITO), sealing (epoxy resin). Performance data:

Thickness: 200-500 nm (SEM, uniformity $\pm 10\%$).

Transmittance change rate: 80%-90% (550 nm, applied voltage ± 2 -4 V).

Response time: 0.5-1.2 seconds for coloring, 0.3-0.8 seconds for fading (electrolyte LiClO₄).

www.chinatungston.cc Cycle stability: 5000-10000 times, transmittance attenuation <5%-8%.

Color efficiency: 50-70 cm²/C.

Apply:

Building energy-saving windows: dimming rate 70%-90%, energy saving 20%-30%, annual demand growth rate of 15%-20%, 2025 is expected to be 10 million to 20 million m².

Automotive anti-glare mirrors: the response time is <1 second, and the annual demand is about 50-1 million pieces.

Display: flexible display (thickness <0.1 mm) with market potential 10-20 tons/year.

4.3.2 Nano-tungsten oxides

AMT prepares WO₃ nanoparticles for use in gas sensors, photocatalysts, and electrochemical devices. The process is:

Spray pyrolysis: AMT solution (WO₃ 20-40 g/L, pH 5.5±0.2) was sprayed into a pyrolysis furnace (600-750°C, N₂ flow rate 400-600 mL/min, residence time 2-5 seconds) through a nozzle (0.5-1 mm).

Collection: Cyclone separator (95%-98% efficiency) or electrostatic capture (voltage 10 kV) to obtain WO3 nanoparticles.

Post-treatment: Ultrasonic cleaning (ethanol, 40-60 kHz, 20-30 minutes), drying (80-100°C, 4-6 hours).

Doping (optional): Add Pt (0.5%-1 wt%) or Pd (0.2%-0.5 wt%) and roast (400°C, 2 hours). .chinatungsten.com Performance data:

Particle size: 10-60 nm (TEM, D50 \approx 20-30 nm).

Specific surface area: $30-50 \text{ m}^2/\text{g}$ (BET).

Gas sensitivity: NO₂ (5-50 ppb, 300°C), response value 50-80, response/recovery time 8-12 seconds; H_2S (1 ppm) with a response value of 30-40.

Photocatalytic efficiency: degradation of methylene blue (20-30 mg/L, UV 365 nm, 2-3 hours), removal rate 90%-98%.

Electrochemical performance: CV cycle (0.1 M H₂SO₄), specific capacitance 200-300 F/g. Apply:

Gas sensors: environmental monitoring (NO₂, CO, H₂S), annual demand of about 100-200 tons. Photocatalysis: wastewater treatment (COD removal rate >90%), air purification (VOCs), market potential 50-150 tons/year.

Electrochemical devices: miniature sensors, flexible electrodes with potential 20-50 tons/year.

4.3.3 Energy storage materials

AMT-derived WO₃ is used in lithium batteries, sodium batteries, and supercapacitor anodes. The process is:

Reduction: AMT is reduced in a tube furnace (500-600°C, $H_2/N_2 = 1:9, 2-4$ hours) to obtain WO₃. Compounding: Mix with carbon black, graphene, or MXene (1:1-1:3 w/w), ball mill (300-500 rpm,

4-8 hours, ball-to-material ratio 10:1).

Electrode preparation: Slurry (WO₃: conductive agent: PVDF = 8:1:1, NMP solvent) coated with copper or aluminum foil, dried (80-100°C, 12-16 hours, thickness 50-100 µm). tungsten.com

Assembly: 2032 button cell (electrolyte 1 M LiPF₆ or NaClO₄).

Performance data:

Lithium battery: capacity 600-750 mAh/g (0.1C, first cycle), 400-500 mAh/g (1C), cycle life 500-

1000 times, capacity retention rate 80%-90%.

Sodium battery: 300-400 mAh/g (0.1C) capacity, 300 cycles, 75%-85% retention.

Supercapacitors: Specific capacitance 250-350 F/g (1 A/g), 10,000 cycles, >90% retention.

Rate performance: 5C, capacity 250-300 mAh/g; 10C, 150-200 mAh/g.

Apply:

Lithium/sodium batteries: electric vehicles (10%-15% increase in range), energy storage systems, annual demand growth rate of 20%-30%, potential 200-500 tons/year.

Supercapacitors: fast charging and discharging equipment (charging time <1 minute), market potential 50-150 tons/year.

4.3.4 Photocatalysts

WO₃ prepared by AMT is used as a photocatalyst for the degradation of organic pollutants and hydrogen production. The process is:

Hydrothermal method: AMT (WO₃ 10-25 g/L) mixed with Na₂SO₄ or NaCl (0.05-0.2 mol/L), hydrothermal autoclave (50-100 mL, 180-220°C, 12-18 hours).

Centrifugation: 8000-12000 rpm, 10-20 min, WO₃.

Roasting: 400-450°C, air, 2-3 hours, enhanced polymorph (monoclinic phase).

w.chinatungsten.com Doping (optional): Ti (1%-3 wt%) or N (0.5%-1 wt%), broaden the photoresponse. Performance data:

Particle size: 20-50 nm (TEM, D50 \approx 30 nm).

Bandgap: 2.5-2.8 eV (UV-Vis, visible light response 400-500 nm).

Photocatalytic activity: rhodamine B (10-20 mg/L, sunlight simulation, 1-2 hours), degradation rate 85%-95%; Hydrogen production rate (10% methanol sacrificial, 300 W Xe lamp), 0.5-1 mmol/h·g. Stability: 5 cycles with a <5% decrease in activity.

Apply:

Water treatment: industrial wastewater (dyes, phenols, COD removal rate 90%-95%), annual demand of about 50-100 tons.

Air purification: VOCs (toluene, formaldehyde) with a removal rate of 80%-90% and a potential of 20-70 tons/year.

Photolysis of water to hydrogen: clean energy, laboratory stage, potential 10-30 tons/year.

4.3.5 Fuel cell electrode materials

AMT-derived WO3 can be used as a proton exchange membrane fuel cell (PEMFC) electrode carrier or co-catalyst. The process is:

Reduction: AMT (550-650°C, $H_2/N_2 = 1:9$, 3-4 hours) yields WO₃.

Composite: mixed with Pt/C (Pt 20%-40 wt%) or Pd/C (Pd 10%-20 wt%) (WO₃:Pt/C = 1:2-1:4), ultrasonic dispersion (ethanol, 40-60 kHz, 1-2 hours).

Electrode preparation: spray (0.2-0.5 mg/cm² Pt) or brush on carbon paper (200 µm thickness) and dry (80-100°C, 4-6 hours).

Assembly: MEA (membrane electrode assembly), Nafion membrane (thickness 50 µm), hot www.chinatun pressing (130°C, 2 MPa).

Performance data:

Electrocatalytic activity: oxygen reduction reaction (ORR), onset potential 0.95-1.0 V (vs. RHE), half-wave potential 0.85-0.90 V, Tafel slope 60-70 mV/dec.

Stability: 5000-10000 CV (0.6-1.2 V, 50 mV/s), activity decay <10%-15%.

Power density: 0.8-1.2 W/cm² (60-80°C, H₂/O₂, pressure 0.1-0.3 MPa).

CO tolerance: 100 ppm CO, activity drop <5%.

Apply:

Fuel cells: new energy vehicles (range 500-600 km), portable power sources, with an annual demand potential of 50-200 tons.

Electrolyzed water: cathode material (HER), market potential 20-50 tons/year.

4.3.6 Thermoelectric materials

AMT-derived WO₃ can be doped to prepare thermoelectric materials that take advantage of their low thermal conductivity and high Seebeck coefficient. The process is:

Synthesis: AMT (WO₃ 20 g/L) and Na₂WO₄ (Na:W = 1:10) were mixed and hydrothermally treated (200°C, 24 hours).

Doping: Add Bi (1%-5 wt%) or Sb (0.5%-2 wt%), ball milling (400 rpm, 6 hours).

Sintering: Spark plasma sintering (SPS, 600-700°C, 50 MPa, 10 minutes) to obtain Na_xWO₃. www.chinatungsten.com Performance data:

Seebeck coefficient: 100-200 µV/K (300-600 K).

Electrical conductivity: 50-100 S/cm.

Thermal conductivity: 1.5-2.0 W/m·K.

ZT value: 0.3-0.5 (500 K).

Density: >98% (theoretical density 7.16 g/cm³).

Apply:

Waste heat power generation: industrial waste heat (300-600°C), recovery efficiency 5%-10%, annual demand potential 20-50 tons.

Miniature thermoelectrics: sensor-powered, market potential 10-30 tons/year.

4.3.7 Electromagnetic shielding materials

AMT-derived WO₃ or W can be used to prepare electromagnetic shielding materials that absorb microwaves and high-frequency electromagnetic waves. The process is:

Reduce: AMT (600-700°C, H₂, 4 hours) to obtain W powder (particle size 0.5-2 µm).

Compounding: Mixed with polymer (PI or PVDF, W: polymer = 70:30-80:20 w/w), hot pressing (200°C, 10 MPa).

Molding: film (0.1-1 mm thick) or coating (spraying, 50-100 µm thick).

Performance data:

Shielding effectiveness: 30-50 dB (1-18 GHz).

Absorption: 80%-90% (10 GHz).

Conductivity: 10²-10³ S/m.

Temperature resistance: 300°C, performance degradation of <5%.

Apply:

Electronic equipment: 5G base station, radar shielding, annual demand of about 50-100 tons.

Aerospace: EMI resistant coatings with a potential of 20-50 tons/year.

4.4 Biomedical applications

4.4.1 Photothermal therapy

AMT-derived WO₃ nanoparticles are used in cancer photothermal therapy due to their near-infrared absorption properties. The process is:

Hydrothermal method: AMT (WO₃ 10-20 g/L) and urea (0.1-0.3 mol/L), 180-220°C, 12-18 hours. Surface modification: PEG (MW 2000-5000, 1%-2 wt%), or HA (hyaluronic acid, 0.5%-1 wt%), stirring (300-400 rpm, 25°C, 4-6 hours).

Centrifugation: 8000-12000 rpm, 10-20 min, WO3 nanoparticles.

Performance data:

Particle size: 15-40 nm (TEM, $D50 \approx 25-30$ nm).

Photothermal conversion efficiency: 38%-45% (808 nm, 1-2.5 W/cm², temperature rise 30-50°C). Biosafety: HeLa, MCF-7 cells, 100-300 µg/mL, 24-48 hours viability>90%-95%.

In vivo distribution: mouse experiment, half-life 6-8 hours, hepatospleen enrichment <20%. Apply:

Tumor treatment: photothermal ablation (80%-90% reduction in tumor volume), laboratory stage, clinical application expected in 2030, annual potential 10-30 tons.

Combination therapy: 30%-50% synergistic effect in combination with chemotherapy drugs (e.g., DOX).

4.4.2 Antimicrobial materials

 WO_3 nanoparticles have photocatalytic antimicrobial properties. The process is the same as above, and the addition is:

Doping: Ag (1%-3 wt%) or Cu (0.5%-2 wt%), enhance antibacterial properties.

Coating: Spray or dip on substrate (glass, metal) with a thickness of 50-100 nm.

Performance data:

Antimicrobial rate: Escherichia coli, Staphylococcus aureus, 99.9%-100% (UV 365 nm, 1-2 hours, concentration 50-100 µg/mL; Ag doping in the dark, antimicrobial rate 80%-90%).

Mechanism: ROS (· OH, O₂⁻) damage cell membranes and DNA.

Durability: 10 cycles, antimicrobial rate >95%.

Apply:

Medical devices: catheters, implant coatings (50%-70% reduction in infection rate), annual demand of about 10-20 tons.

Antimicrobial textiles: surgical gowns, masks, market potential 5-15 tons/year.

Utilities: door handles, elevator button coatings, potential 10-20 tons/year.

4.4.3 Drug delivery vehicles

WO₃ nanoparticles can be used as drug delivery carriers due to their porous structure. The process is:

Synthesis: AMT (WO₃ 20-30 g/L) and CTAB (0.05-0.1 mol/L), hydrothermal (180-200°C, 12-16

hours).

Loading: Doxorubicin (DOX, 10-20 mg/mL) or Cisplatin (5-10 mg/mL), impregnation (25-37°C, 24-36 hours, 20%-30% loading).

Coating: Polydopamine (PDA, 1-2 mg/mL) or SiO₂ (TEOS, 0.5 mL), enhance stability.

Performance data:

Pore size: 5-15 nm (BET, pore volume 0.2-0.3 cm³/g).

Release rate: pH 5.0 (tumor microenvironment), 75%-85% release in 48 hours; pH 7.4 (blood), release <15%-20% in 24 hours.

Cytotoxicity: MCF-7, A549 cells, IC50 \approx 3-6 µg/mL (containing DOX), targeting increased by 20%-30%.

Stability: 4°C, 3 months, drug leakage <5%.

Apply:

Cancer therapy: targeted delivery (tumor enrichment rate 10%-15%), laboratory stage, potential 5-15 tons/year.

Chronic disease: sustained-release insulin with a potential of 2-5 tons/year.

4.4.4 Bioimaging probes chimatung

WO₃ nanoparticles doped with rare earth elements (e.g., Eu^{3+} , Tb^{3+}) as fluorescent probes. The process is:

Synthesis: AMT (WO₃ 15-25 g/L) with Eu(NO₃)₃ (5%-10 mol%) or Tb(NO₃)₃ (3%-8 mol%), hydrothermal (200-220°C, 16-20 hours).

Modification: Silanization (APTES, 1%-2 wt%) or PEG (MW 2000, 0.5%-1 wt%) to improve water solubility.

Centrifugation: 10,000-15,000 rpm, 15-20 min.

Performance data:

Particle size: 30-60 nm (TEM, $D50 \approx 40$ nm).

Fluorescence: Eu^{3+} (excitation 394 nm, emission 615 nm, red), quantum yield 15%-25%; Tb³⁺ (excitation 378 nm, emission 545 nm, green) with 20%-30% yield.

Biological Safety: L929 cell viability >95%-98% at concentrations of 50-100 μ g/mL with no significant toxicity.

Imaging depth: 5-10 mm tissue penetration (near-infrared excitation).

Apply:

Cell imaging: cancer cell labeling (50%-80% increase in fluorescence intensity), laboratory stage, potential 2-10 tons/year.

In vivo imaging: small animal experiments (resolution 0.1-0.5 mm) with potential 1-5 tons/year.

4.4.5 Bone repair materials

WO₃ nanoparticles can be compounded with bioceramics to promote bone regeneration. The process is:

Synthetic: AMT (WO₃ 20 g/L) with Ca(NO₃)₂ (Ca:W = 1:1), hydrothermal (180°C, 12 h). Compound: Mixed with hydroxyapatite (HA, 50%-70 wt%), ball mill (300 rpm, 4 hours). Forming: pressing (150 MPa), sintering (1000-1100°C, 2 hours) to obtain WO₃/HA composites.

Performance data:

Porosity: 30%-40% (SEM, pore size 100-500 μm).

Compressive strength: 50-80 MPa.

Biological activity: MC3T3-E1 osteoblasts, 7-day proliferation rate increased by 30%-50%, ALP hinatungsten.com activity increased by 40%.

Degradation rate: <5% mass loss at 28 days in PBS.

Apply:

Bone defect repair: implants (bone filling rate 60%-80%), laboratory stage, potential 5-15 tons/year. Dental materials: Dental implant coating with potential 2-5 tons/year.

4.4.6 Antioxidants

WO₃ nanoparticles scavenge free radicals due to their antioxidant properties. The process is: Synthesis: AMT (WO₃ 15 g/L) and H₂O₂ (0.1 mol/L), hydrothermal (160°C, 10 hours). Modification: Chitosan (0.5%-1 wt%) for enhanced dispersion.

Particle size: 20-40 nm (TEM). Antioxidant capacit Antioxidant capacity: DPPH free radical scavenging 70%-85% at concentration 100 ug/mL, superoxide anion (O₂⁻) scavenging 60%-75%.

Biosafety: Concentrations of 50-200 µg/mL, cell viability >95%, no inflammatory response.

Apply:

Nutraceuticals: anti-aging additives, laboratory stage, potential 2-10 tons/year.

Cosmetics: antioxidant skincare (30%-50% increase in free radical protection) with a potential of 5-15 tons/year.

4.5 Market and Prospects

4.5.1 Global Market Overview

AMT produces about 8,000-12,000 tons per year (China accounts for 70%-75%), and is mainly exported to Japan and South Korea (electronic materials, 30%-35%), Europe and the United States (catalysts, tungsten, 45%-50%), and other regions (10%-15%). The market size will be about 2 billion to 3 billion yuan in 2023, with an annual growth rate of 5%-10%. Market Segments: Traditional tungsten products: 55%-65% (5000-7000 tons), annual growth of 3%-5%.

Catalysts: 20%-25% (2000-3000 tons), 5%-8% increase.

Emerging materials: 10%-15% (1000-1500 tons), 15%-25% increase.

Biomedical: <5% (50-200 tons), growth potential 20%-30%.

4.5.2 Challenges and development directions

Challenge:

Purity requirements: impurities (Fe, Mo) < 0.0005%, and the purification process needs to be improved.

Environmental pressure: ammonia nitrogen emission <10 mg/L, waste liquid treatment cost

increased by 10%-20%.

Cost competition: RMB 2-35,000 per ton, reduced by 5%-15%.

Technical barriers: nanoscale application consistency (particle size deviation <10%). Direction:

Green process: microwave synthesis (20%-30% reduction in energy consumption), waste liquid recycling (ammonia recovery rate >95%).

Intelligent: Automated production line (10%-15% more consistency), AI-optimized process parameters.

Emerging applications: nanotechnology (sensors, batteries, 20% annual growth), biomedical (photothermal, imaging, potential 100-500 tons/year).

International cooperation: Deepen technical exchanges with Japan and South Korea (electronics), Europe and the United States (military industry), and increase the proportion of exports to 50%.

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Ammonium metatungstate product introduction

1. Product Overview

Ammonium metatungstate (AMT) with the chemical formula (NH₄)₆H₂W₁₂O₄₀·xH₂O, is a highly soluble tungsten compound with a white or yellowish crystalline powder. AMT is an important intermediate raw material for the production of tungsten products and other tungsten compounds, and is widely used in many industrial fields due to its excellent water solubility (solubility up to 303.9g/100g H₂O at 20°C) and thermal stability.

Second, product characteristics

Appearance: White or yellowish crystalline powder

Purity: ≥99.95%

Solubility: High water solubility, insoluble in ethanol

Density: approx. 2.3 g/cm³

Thermal stability: decomposes into tungsten trioxide (WO₃) above 300°C

Safety: It is slightly acidic and irritating, so you need to pay attention to protection when using it tungsten.

3. Product specifications

WO₃ 含量 (≥% min) 91.0										
Impurity co	Impurity content (max., %)									
element	To the	As	Bi	Ca	With	Fe	Mg	Κ	Mn	Мо
maximum	0.0010	0.0010	0.0001	0.0010	0.0005	0.0020	0.0005	0.0010	0.0010	0.0030
element	On	Nor	Р	Pb	S	Sb	Yes	Sn	Ti	V
maximum	0.0020	0.0005	0.0007	0.0010	0.0030	0.0005	0.0010	0.0010	0.0010	0.0010

4. Packaging and warranty

Packing: Inner sealed vacuum plastic bag, outer iron drum or plastic drum, net weight 50kg, moisture-proof and anti-oxidation.

Warranty: With quality certificate, tungsten content, impurity analysis (ICP-MS), particle size (FSSS method), loose density and moisture data, shelf life of 12 months (sealed and dry conditions).

5. Procurement information

Mailbox:sales@chinatungsten.com Phone: +86 592 5129696

For more information about ammonium metatungstate, please visit China Tungsten Online (www.ammonium-metatungstate.com). www.chinatungsten

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Chapter 5 Future Developments and Challenges

Ammonium metatungstate (AMT, chemical formula (NH₄)₆H₂W₁₂O₄₀·nH₂OAs an important intermediate of tungsten chemical industry, it has shown great potential in traditional industries, emerging functional materials and biomedical fields. However, with the growing global demand for high-performance materials, stricter environmental regulations, and accelerated technological innovation, the development of AMT faces many opportunities and challenges. This chapter discusses the future development direction of AMT from four aspects: technological innovation, market trends, environmental protection and sustainable development, and industrialization bottlenecks, and proposes coping strategies to provide guidance for researchers and industry.

5.1 Technological innovation

5.1.1 Green synthesis technology

The traditional preparation methods of AMT (such as acidification method and ion exchange method) have problems such as high energy consumption and large waste liquid discharge, and green synthesis technology has become the focus of the future.

Microwave-assisted synthesis: Microwave heating (2450 MHz, power 800-1000 W) reduces reaction time from 1-2 hours to 10-15 minutes and reduces energy consumption by 20%-30%. Experimental data: yield 88%-93%, WO₃ content 90%-91%, particle size 1-5 μm.

Ultrasonic-assisted extraction: Ultrasonic (40 kHz, 200 W) enhances solvent extraction efficiency (TBP/kerosene) with a 50% reduction in extraction time (20-30 minutes), a yield increase of 92%-96%, and a 15%-20% reduction in waste.

Biotechnology: Extraction of tungstate from tungsten ore using microorganisms (e.g., acidophilus)

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and conversion to AMT with 70%-80% yield and 40% lower energy consumption in the laboratory phase, but with a long reaction cycle (5-7 days).

Development Trends:

Develop low-cost, high-efficiency microwave/ultrasonic equipment (investment of 30-500,000 yuan/set) to achieve industrial amplification (annual output of 1000-2000 tons).

Combine AI to optimize reaction parameters (e.g., temperature, pH) and improve consistency (yield fluctuations of <2%).

5.1.2 Nanoization and functionalization

AMT-derived WO₃ or W nanomaterials are surging in demand in sensors, batteries, and biomedicine, driving the development of nano- and functionalization technologies.

Nanoization: Hydrothermal method (180-220°C, 12-18 hours) combined with ultrasonic dispersion (60 kHz, 30 min) to prepare WO₃ nanoparticles (particle size 10-30 nm, D50 \approx 20 nm) with a specific surface area of 40-60 m²/g.

Functionalization: Surface modifications (e.g., PEG, SiO₂) enhance WO₃ water solubility (50%-80% solubility) and doped Pt (0.5%-2 wt%) or Eu³⁺ (5%-10 mol%) to improve catalytic or fluorescence performance.

Examples: Pt-doped WO₃ nanoparticles were used in NO₂ sensors (sensitivity 10 ppb, response time <5 seconds), and Eu³⁺-doped WO₃ was used for bioimaging (60% increase in fluorescence intensity). Development Trends:

Development of continuous nanofabrication equipment (e.g., microfluidic reactor with >95% yield) to meet annual demand of 100-500 tons.

Functionalization technology is moving towards multifunctional composites (e.g. WO₃/graphene, 20%-30% capacitance).

5.1.3 Intelligent production

Smart manufacturing technologies, such as Industry 4.0, will optimize AMT productivity and quality control.

On-line monitoring: real-time monitoring of pH, temperature and concentration (accuracy ± 0.05), PLC system automatically adjusts the addition of acid and alkali (error <0.1), reducing manual operation by 30%-50%.

Big data analytics: Reaction data (10⁴-10⁵ data points/batch) is collected and AI predicts the optimal parameters (e.g., pH 5.5±0.1, yield increase by 5%-8%).

Case: A tungsten company in China introduced an intelligent production line (with an investment of 5 million yuan) with an annual output of 2,000 tons of AMT, reducing waste liquid discharge by 25% and labor costs by 40%.

Development Trends:

Promote intelligent equipment (cost 1 million to 3 million yuan/set), realize the automation of the whole process, and increase the annual production efficiency by 15%-20%.

Develop digital twin technology to simulate the production process and reduce the cost of trial and error by 20%-30%.

5.2 Market Demand and Trends

5.2.1 Upgrading of traditional fields

The demand for AMT in traditional fields such as tungsten carbide and tungsten is stable, but the demand for high-purity, small-particle size products is increasing.

High-purity tungsten powder: the purity requirement is increased from 99.9% to 99.99%, and the impurities (Fe, Mo) < 0.0005% for semiconductor targets (film thickness uniformity ±3%).

Fine-grained tungsten powder: Particle size from 1-5 μ m to 0.1-1 μ m (D50 \approx 0.5 μ m) for 3D printing (part accuracy \pm 0.05 mm), with an annual demand increase of 10%-15%.

Market data: The global demand for tungsten powder will be about 50,000 tons in 2023, with AMT accounting for 60%-70% (3000-3500 tons), and it is expected to reach 60,000 tons in 2030. Development Trends:

Development of ultra-fine tungsten powder preparation technology (e.g. plasma reduction, particle size $<0.5 \mu m$) to meet the needs of high-end manufacturing.

The proportion of AMT exports has increased from 40% to 50%-60%, and the target markets are Japan and South Korea (electronics), Europe and the United States (military industry).

5.2.2 Growth in emerging areas

Emerging fields (e.g., batteries, sensors, biomedicine) are experiencing a rapid increase in demand for AMT.

Energy storage: Lithium/sodium battery anode WO₃ demand is expected to increase from 50 tonnes/year (2023) to 500-1000 tonnes/year (2030), with an annual growth rate of 20%-30%.

Sensors: WO_3 nanoparticles for NO_2 , H_2S detection, annual demand increased from 100 tons to 300-500 tons, a growth rate of 15%-20%.

Biomedical: Photothermal therapy, drug delivery WO_3 demand increased from <10 tonnes (laboratory) to 50-200 tonnes (clinical phase), with a growth potential of 25%-35%.

Case: A Chinese company exported 50 tons of WO₃ nanoparticles to the United States for battery research and development in 2023, with a contract value of 20 million yuan.

Development Trends:

Customized AMT products for emerging fields (e.g., nanoscale, high activity) increased market share from 10% to 20%-25%.

Strengthen cooperation with new energy and medical enterprises, and lock in long-term orders (annual supply of 500-1000 tons).

5.2.3 Regional Market Differences

The market demand for AMT varies significantly due to regional economic development and technological level.

China: 70%-75% (6,000-8,000 tons) of global production, mainly used for tungsten (60%), catalysts (25%), and emerging fields account for <15%, with a growth rate of 5%-8%.

Japan and South Korea: demand 1000-1500 tons, electronic materials (50%), sensors (30%), growth rate of 10%-15%.

Europe and the United States: demand 1500-2000 tons, military industry (40%), catalyst (35%),

growth rate of 5%-10%.

Developing countries: demand < 500 tonnes, infrastructure construction drives tungsten demand, with growth potential of 15%-20%.

Development Trends:

China's transition to high value-added products (from 5% to 20% of nanomaterials).

Japan, South Korea, Europe and the United States have deepened technical cooperation to develop customized AMT (such as doped type).

5.3 Environmental protection and sustainable development

5.3.1 Waste liquid treatment

AMT production produces ammonia nitrogen waste liquid (5-15 g/L) and acidic wastewater (pH 2-4), which exacerbates environmental pressure.

Status: The cost of the traditional neutralization method $(Ca(OH)_2)$ is 500-800 yuan/ton of waste liquid, and the recovery rate of residue $(CaWO_4)$ is 50%-60%.

Improved technology:

Ammonia distillation recovery: ammonia distillation tower (steam pressure 0.4-0.6 MPa), ammonia recovery rate is 90%-95%, concentration is 10%-15%, and the cost is reduced to 300-500 yuan/ton. Membrane separation: nanofiltration membrane (molecular weight cut-off 200-400 Da), waste liquid concentration 5-10 times, recovery of WO₃ 80%-90%, investment of 50-1 million yuan/set.

Case: In 2022, a factory in Jiangxi Province will use ammonia distillation + membrane separation, with an annual processing capacity of 5,000 tons of waste liquid, 200 tons of ammonia recovery, and 50 tons of WO₃ recovery, saving 30% of costs.

Development Trends:

Promote zero-emission technology, and reduce ammonia nitrogen emissions to <5 mg/L (better than China's GB 8978-1996 standard).

The recycling rate of waste liquid has increased from 50% to 80%-90%.

5.3.2 Energy consumption and carbon emissions

AMT production has high energy consumption (roasting, crystallization, etc.), and carbon emissions need to meet global emission reduction targets.

Status: Energy consumption of 2000-3000 kWh per ton of AMT, CO₂ emissions of 1.5-2.0 tons (mainly coal-fired power).

Improved technology:

Renewable energy: Solar (photovoltaic power 100-200 kW) or wind power, with 20%-30% lower energy consumption and 30%-40% reduction in carbon emissions.

Waste heat recovery: Roaster exhaust gas (300-500°C) drives the steam turbine to recover 15%-20% of the heat, saving 50-1 million kWh of electricity per year.

Case study: A company will introduce waste heat recovery in 2023, with an annual output of 1,000 tons of AMT, saving energy consumption by 15% and reducing CO₂ emissions by 300 tons.

Development Trends:

Carbon emissions will be reduced to 1.0-1.2 tons/ton AMT in 2030, in line with the goal of carbon

neutrality.

Energy consumption is optimized to 1500-2000 kWh/ton, and the proportion of clean energy is increased to 50%.

5.3.3 Resource recycling

Tungsten resources are limited (about 3.4 million tonnes of global reserves, 60% of which are in China), and recycling is key to sustainable development.

Status: 30%-50% tungsten recovery (scrap carbide, catalyst), 5%-10% tungsten loss in AMT production.

Improved technology:

Waste tungstic acid recovery: WO_3 (1-5 g/L) in the waste liquid is passed through ion exchange (D001 resin) with a recovery rate of 85%-95%.

Scrap alloy recycling: Tungsten powder (>99% purity) is recovered by electrolysis (current density $100-200 \text{ A/m}^2$) with an annual processing capacity of 500-1000 tons.

Example: A German company recycles 300 tons of tungsten scrap and prepares 200 tons of AMT in 2022, reducing the cost by 20%.

Development Trends:

Tungsten recycling rate increased from 50% to 70%-80%.

Establish a global tungsten resource recovery network to reduce raw ore mining by 10%-15%. www.chi

5.4 Industrialization bottlenecks and solutions

5.4.1 Cost control

The production cost of AMT (2-35,000 yuan/ton) is high in the competition in emerging fields. Bottleneck: raw material (tungsten concentrate 10-150,000 yuan/ton), energy (500-800 yuan/ton), labor (300-500 yuan/ton) accounted for a high proportion.

Solution:

Feedstock substitution: Utilizing low-grade tungsten ore (WO₃ 20%-30%), flotation purification to 95%, cost reduction of 10%-15%.

Automation: Reduce labor by 40%-50%, and save 200-300 yuan per ton cost.

Scale: Increase annual production from 1,000 tons to 5,000 tons, and reduce unit costs by 15%-20%.

Emerging applications require strict AMT purity and particle size consistency. WWW.chimatum Bottlenecks: 1%-2% fluctuation in WO: contact Bottlenecks: 1%-2% fluctuation in WO₃ content and 10%-20% particle size deviation between batches.

Solution:

Precise control: on-line monitoring (pH ± 0.05 , temperature $\pm 1^{\circ}$ C), consistency is improved to more than 99%.

Standardization: Develop AMT quality standards (e.g. WO₃ >90%, Fe <0.0005%) in line with ISO.

5.4.3 Technical Barriers and Competition

The technical barriers in emerging fields are high, and international competition is intensifying.

Bottlenecks: European and American companies are leading in the patent layout in the fields of nanomaterials and biomedicine, while Chinese companies have insufficient technical reserves. Solution:

R&D investment: The proportion of enterprise R&D has increased from 3%-5% to 8%-10%, with an annual investment of 50 million to 80 million yuan.

Industry-university-research cooperation: joint development of nano-WO₃ with universities (e.g. Tsinghua University) (10-20 patent applications/year).

Patent layout: Focus on the protection of green technology and functional technology, and the number of patents has increased from 50 to 200.

5.4.4 Policies and Regulations

Environmental protection and trade policies affect the industrialization of AMT.

Bottlenecks: China's environmental protection tax (1000-2000 yuan/ton), European and American anti-dumping duties (10%-20%).

Solution:

Compliant production: Waste liquid discharge meets the standard (ammonia nitrogen <10 mg/L), and strives for tax deduction of 20%-30%.

Diversified markets: Expand the market in Southeast Asia and Africa, and increase the proportion of exports from 5% to 15%-20%.

Resources

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Chapter 6 Industrial Production and Technological Challenges

Ammonium metatungstate (AMT, chemical formula (NH4)6H2W12O40 nH2O) is an important intermediate of tungsten chemical industry, which is widely used in traditional tungsten products, emerging functional materials and biomedical fields. However, the scale of its industrial production faces technical bottlenecks such as purity control and process efficiency, and it needs to improve green processes, intelligent technologies and safety and environmental protection measures. This chapter systematically discusses the challenges and improvement directions of large-scale production of AMT from the perspective of technology and process, and provides technical reference for achieving efficient and sustainable production. ww.chinatungsten.com

6.1 Bottlenecks in large-scale production

6.1.1 Purity control

The purity of ammonium metatungstate (AMT) directly affects the quality of downstream products, but it is difficult to maintain consistency in large-scale production.

Status: The WO₃ content of AMT can reach 90%-91% under laboratory conditions, but fluctuates between industrial production batches by 88%-90%, and the content of impurities (e.g., Fe, Mo, Na) increases from <0.01% to 0.02%-0.05%.

Reasons for the purity of ammonium metatungstate (AMT):

Raw material fluctuations: Tungsten concentrate WO₃ content varies (60%-75%), and impurities (e.g. Fe 0.5%-2%) are difficult to remove completely.

Process scale-up: The reactor (500-1000 L) was stirred unevenly, and the pH control deviation $(\pm 0.2-0.5)$ led to inconsistent polytungstate polymerization.

Crystallization process: fluctuating cooling rate (0.5-2°C/min), crystal inclusion of impurities.

Purity effects of ammonium metatungstate (AMT):

The purity decreased from >99.95% to 99.9%, affecting the uniformity of the semiconductor target film thickness ($\pm 5\%$ to $\pm 10\%$).

inatungsten.com Reduced catalyst activity (NO_x conversion from >95% to 85%-90%).

Ammonium metatungstate (AMT) purity solutions:

Raw material pretreatment: Tungsten concentrate is purified by flotation and magnetic separation to WO₃ >97%, Fe <0.02%.

Precise control: Purity fluctuations are reduced to <0.5% using an in-line pH meter (accuracy ± 0.05) and optimized agitation (400-600 rpm).

Fractional crystallization: secondary crystallization (5°C, cooling rate 0.2°C/min) is implemented, and the impurity removal rate is increased by 20%-30%.

6.1.2 Energy consumption and efficiency

Energy consumption and efficiency issues in ammonium metatungstate (AMT) production limit large-scale applications.

Status: 2,000-3,000 kWh per tonne of AMT, mainly in roasting (50%), concentration (30%) and crystallization (15%).

Cause:

Low-grade feedstock: Tungsten ore with a WO₃ content of <70% needs to be purified multiple times, increasing energy consumption by 20%-30%.

Conventional equipment: The thermal efficiency of the rotary kiln is only 60%-70%, and the heat loss of the evaporator is 30%-40%.

Complex process: Multi-step reactions (e.g., acidification, concentration, crystallization) are inefficient and yield fluctuations range from 85% to 90%.

Effect:

High energy consumption extends the production cycle (12-24 hours per batch), making it difficult to meet the demand for large batches.

Inefficiency leads to poor batch consistency, affecting downstream product stability.

Case: In 2023, due to the low efficiency of the equipment, the batch cycle was extended to 20 hours, and the yield was only 87%.

Solution:

Raw material optimization: high-grade tungsten ore (WO₃ > 80%) is selected, and the purification step is reduced, and the energy consumption is reduced by 15%-20%.

High-efficiency equipment: The introduction of a thin-film evaporator (>85% efficiency) and a waste heat recovery system reduced energy consumption to 1500-2000 kWh/ton.

Process simplification: Optimized concentration and crystallization integrated process, the production cycle time is shortened to 10-15 hours, and the yield is increased to 90%-93%. www.chinatun

6.2 Direction of technical improvement

6.2.1 Green synthesis process

Traditional processes (such as acidification) produce a large amount of waste liquid and high energy consumption, and green synthesis technology is the focus of improvement.

Status: The yield of acidification method is 85%-90%, the ammonia nitrogen waste liquid is 5-10 g/L, and the energy consumption is 2000-2500 kWh/ton.

Improved technology:

Microwave synthesis: Using a microwave reactor (2450 MHz, power 800-1200 W), the reaction time is reduced from 1-2 hours to 10-20 minutes, the energy consumption is reduced to 1500-1800 kWh/ton, and the WO₃ content is 90%-91%.

Ultrasonic extraction: Ultrasonic (40 kHz, power 200-300 W) assisted solvent extraction (TBP 30%), extraction efficiency increased by 15%-20%, waste volume reduced by 20%-30%, WO₃ recovery rate >90%.

Bioleaching: Acidophilus (e.g., Thiobacillus ferrooxidans) extracts tungstate from low-grade tungsten ore with a yield of 70%-80%, a 30%-40% reduction in energy consumption, and a reaction cycle of 5-7 days.

Advantage:

Microwave synthesis: the particle size uniformity is increased by 10%-15%, which is suitable for the preparation of nanomaterial precursors.

Ultrasonic extraction: The ammonia nitrogen in the waste liquid is reduced to 3-5 g/L, and the degree of greening of the process is improved.

Case: A factory piloted microwave synthesis (50 kg/batch) in 2022, reducing energy consumption by 25%, waste liquid by 20%, and yielding 92%.

Development Trends:

Promote microwave and ultrasonic equipment to achieve large-scale application with an annual output of 2000-5000 tons.

The bioleaching scale-up process was developed to shorten the reaction cycle to 2-3 days and increase the yield to 85%-90%.

6.2.2 Automation and intelligent production

Automation and intelligence increase productivity and quality consistency.

Status: Traditional production relies on manual operations (50%-70%), with batch consistency fluctuating by 2%-5%.

Improved technology:

Automation: The reactor (500-1000 L) is equipped with a metering pump (acid-base flow rate 2-5 L/h) and an online monitoring system (pH accuracy ± 0.05 , temperature $\pm 1^{\circ}$ C), reducing manual operation by 40%-60%.

Intelligent: The PLC system controls the process parameters, and the big data analysis optimizes the pH (5.5 ± 0.1) and temperature ($80\pm1^{\circ}$ C), and the yield is increased by 5%-10%.

Digital twin: Predict reaction progress with simulation software such as Aspen Plus, optimize energy

consumption and yield, and reduce trial-and-error time by 20%-30%.

Advantage:

Automation: WO₃ content fluctuations are reduced to <0.5% and batch consistency is improved to more than 99%.

Intelligent: The stability of equipment operation is improved, and the failure rate is reduced by 15%-20%.

Case: A company introduced an automated production line (with an annual output of 1,000 tons) in 2021, with a consistency of 99.5% and a 15% reduction in energy consumption.

Development Trends:

Promote full-process automation equipment to achieve an annual production efficiency increase of 20%-30%.

Developed a digital platform for AMT production to monitor process parameters in real-time (synchronization error of <1 second) to support global plant collaboration.

6.3 Safety and Environmental Protection

6.3.1 Safety specifications in the production process

The production of ammonium metatungstate (AMT) involves high temperature, high pressure and chemical reagents, and requires strict safety management. Status:

High temperature risk: Roasting (800-900°C), concentration (80-100°C) may cause burns or fire. Chemical risk: volatilization of ammonia (25%-28%), HCl (2-3 mol/L), inhalation or contact hazards.

Equipment risk: Excessive reactor pressure (0.1-0.5 MPa) may lead to explosion.

Specification:

Equipment safety: The reactor is equipped with a pressure safety valve (limited pressure of 0.6 MPa) and a temperature alarm system (triggered > 100° C), and the failure rate is reduced to <1%.

Personnel protection: Operators are equipped with acid-proof suits and gas masks (filtration efficiency >95%), regular medical examinations (2 times a year).

Emergency plan: ammonia leakage with neutralizer (NaOH reserve 500 kg), fire drill (1 time per quarter).

Example: A factory stopped production for 3 days in 2022 due to an ammonia leak (0.5 m³), and the WWW.chine accident rate dropped to 0 after improvement.

Development Trends:

AMT production safety standards (refer to OSHA or GB/T 13869) were established to reduce the accident rate to <0.5%.

Promote an intelligent early warning system (NH₃ concentration >0.1 ppm alarm) and reduce response time to <5 seconds.

6.3.2 Waste liquid and waste gas treatment

Liquid and exhaust gases from AMT production need to be treated efficiently to meet environmental www.chinatung requirements.

Status quo:

Effluent: ammonia nitrogen 5-15 g/L, WO₃ 1-5 g/L, pH 2-4, annual discharge 5000-10000 tons (annual output of 1000 tons).

Exhaust gases: NH₃ 0.5-1.5 g/m³, VOCs (ethanol, etc.) 0.2-0.5 g/m³, annual emissions 10⁴-10⁵ m³. Handling technology:

Liquid Waste Treatment:

Ammonia recovery: Ammonia evaporation tower (steam pressure 0.4-0.6 MPa), ammonia recovery rate 90%-95%, residual liquid ammonia nitrogen <15 mg/L.

Membrane separation: nanofiltration membrane (molecular weight cut-off 200-400 Da), WO₃ recovery 85%-90%, and 5-10-fold concentration of waste.

Exhaust gas treatment:

Pickling tower: H₂SO₄ (5%) absorbs NH₃, discharges <0.1 g/m³, efficiency >95%.

Activated carbon adsorption: VOC removal, emission $<0.05 \text{ g/m}^3$, adsorption capacity 100-150 g/kg. Example: A factory will process 6,000 tons of waste liquid in 2023, recover 150 tons of ammonia and 40 tons of WO₃, and meet the emission standards (ammonia nitrogen <10 mg/L).

Impact: Untreated effluent leads to soil acidification (pH drop of 0.5-1) and 80% reduction in environmental impact after improvement.

Development Trends:

Achieve a zero-emission target (ammonia nitrogen <5 mg/L, WO₃ recovery rate >95%). Promote exhaust gas heat recovery technology, saving energy consumption by 10%-15%.

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Chapter 7 Case Studies and Technical Analysis

Ammonium metatungstate (AMT, chemical formula $(NH_4)_6H_2W_{12}O_{40}\cdot nH_2O$) is the core intermediate in the field of tungsten chemical industry, and its preparation process and application technology directly affect the quality of downstream products. This chapter systematically discusses the technical practice and optimization direction of AMT through industrial production cases (highpurity AMT and catalyst applications), laboratory synthesis examples, and failure analysis, so as to provide reference for industrial production and scientific research.

7.1 Industrial production cases

7.1.1 Preparation examples of high-purity AMT

Background: A tungsten company in Hunan, China, has developed high-purity AMT (WO₃ >91%, impurity <0.005%) to meet the demand for tungsten powder for semiconductor targets, with an annual output target of 1500 tons.

Process:

Raw material treatment: wolframite (WO₃ 68%) is purified to 98% by flotation, and tungstic acid is obtained by roasting (900°C, rotary kiln, 6 hours).

Dissolution and adjustment: Tungstic acid was dissolved in aqueous ammonia (25%, 80°C, stirring at 300 rpm, 2 h), and HCl (2 mol/L, flow rate 2 L/h) was added dropwise to pH 5.5±0.1.

Concentration and crystallization: Concentration in a thin-film evaporator (80°C, pressure 0.08 MPa), cooling to 5°C (rate 0.2°C/min), crystallization for 12 hours.

Post-treatment: centrifugation (5000 rpm, 10 min), drying (100°C, 4 h), AMT.

Technical Parameters:

WO₃ content: 91.2%-91.5% (titration).

Impurity content: Fe <0.002%, Mo <0.001%, Na <0.002% (ICP-AES).

Yield: 92%-94% (based on WO₃).

Particle size: $D50 \approx 3-5 \,\mu m$ (laser particle size analysis).

Technical Highlights:

A high-precision pH meter (± 0.05) and two-step crystallization (one to remove 80% of impurities and a second to improve purity) were used to ensure a WO₃ content of >91%.

The thin-film evaporator increases concentration efficiency (>85%) and reduces energy consumption by 15%-20%.

Application: Preparation of tungsten powder (purity >99.98%, $D50 \approx 1 \mu m$) for sputtering targets (film thickness uniformity $\pm 3\%$).

7.1.2 Application examples of AMT for catalysts

A European chemical company uses AMT to produce WO₃/V₂O₅/TiO₂ denitrification catalysts, which meet the NO_x emission standard of power plant boilers ($\leq 50 \text{ mg/Nm}^3$) with an annual output chinatungsten.coi of 1200 tons.

Process:

Preparation of AMT: Sodium tungstate (WO₃ 120 g/L) was ion exchanged (Amberlite IR-120, flow rate 2 BV/h), pH 2-3, ammonia water was adjusted to pH 5.5, concentrated and crystallized to obtain AMT (WO₃ 90%-91%).

Catalyst preparation: AMT solution (WO₃ 60 g/L) impregnated with TiO₂ (specific surface area 90 m²/g, WO₃ loading 10 wt%), ultrasonic dispersion (40 kHz, 30 min), drying (120°C, 6 h), roasting (550°C, 4 h); Add NH4VO3 (V2O5 2 wt%) and re-roast (480°C, 3 hours).

Forming: Extruder molding (honeycomb, pore density 400 cpsi), cutting (50×50×100 mm).

Technical Parameters:

AMT yield: 88%-90%.

www.chinatungs Catalyst performance: NO_x conversion >96% at 300°C (GHSV 40,000 h⁻¹).

Specific surface area: 65-70 m²/g (BET).

Antitoxicity: SO₂ (1000 ppm, 48 hours), <5% decrease in activity.

Technical Highlights:

Ultrasonic impregnation ensures uniform WO₃ distribution with a load deviation of <5%.

Two-stage roasting optimizes the crystal form (monoclinic WO₃) and increases the catalytic activity by 10%-15%.

Application: Used for denitrification in coal-fired power plants, NO_x removal rate >95%, life 2-3 years.

7.2 Examples of laboratory synthesis

7.2.1 Small-scale experimental design

A university laboratory designed an AMT synthesis experiment with the goal of preparing 100 g of high-purity AMT (WO₃ >90%) for nano-WO₃ research.

Experimental Procedure:

Raw material preparation: Tungstic acid (WO₃ 98%, 10 g) dissolved in 50 mL of ammonia (25%), stirred (400 rpm, 60°C, 1 hour).

Acidification: Add HNO₃ (1 mol/L, flow rate 1 mL/min) dropwise to pH 5.5±0.1, and the solution becomes clear.

Concentration and crystallization: Concentrate in a water bath (80°C, 2 hours, 0.09 MPa decompression) and cool to 5°C (ice bath, 6 hours).

Post-treatment: Filtration (0.45 µm membrane), drying (100°C, 3 hours).

Technical Parameters:

WO₃ content: 90.5%-91% (titration).

ungsten.com Yield: 85%-88% (based on WO₃).

Impurities: Fe <0.005%, Na <0.003% (ICP-MS).

Crystal morphology: needle-shaped (SEM), particle size 5-10 µm.

Technical Highlights:

The small-volume reactor (100 mL) ensures pH control accuracy (± 0.05).

Ice bath crystallization slows down the cooling rate (0.1°C/min) and improves crystal purity.

7.2.2 Data Analysis and Optimization

Data analysis:

Yield influencing factors: highest yield at pH 5.5 (88%), 10%-15% decrease at pH <5 or >6 (polytungstate decomposition or non-conversion).

Purity effect: WO₃ content at 25% ammonia concentration 90.5% and down to 89% at 30% (increased Na⁺ residue).

Experimental repeatability: WO₃ content fluctuated by <0.5% and yield fluctuated by <2% in 3 experiments.

Optimization Solution:

pH optimization: fine-tuning to 5.4-5.6, using a buffer (NH₄Cl, 0.1 mol/L), the yield increased to 90%-92%.

Concentration improvements: Microwave heating (800 W, 10 minutes) instead of water baths reduces concentration time by 50% and increases WO₃ content to 91%-91.5%.

Results: The optimized post-yield was 91% and the purity was 91.3%, which was suitable for the preparation of nanomaterial precursors.

7.3 Failure Analysis and Solutions

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7.3.1 Frequently Asked Questions

Poor crystallization:

Manifestations: Crystals are small (<1 µm) or crystalline, and the solution is turbid.

Causes: pH deviation (<5 or >6), polytungstate decomposition to $WO_{4^{2^{-}}}$ or excessive polymerization. The cooling rate is too fast (>1°C/min), and the crystal nuclei are excessive. Impurities (e.g., Ca²⁺, Mg²⁺ >0.01%) inhibit crystal growth.

Impact: 20%-30% reduction in yield, WO3 content <88%...atume

Impurities exceed the standard: Fe >0.02%, Mo >0.01%, Na >0.02%.

Causes: Insufficient purity of raw materials (Fe tungsate >0.05%), incomplete cleaning of the reactor, residual metal ions, ungraded crystallization process, impurity entrapment.

Impact: Downstream tungsten powder purity < 99.9%, catalyst activity reduced by 10%-15%.

7.3.2 Resolution Strategy

Poor crystallization:

pH control: In-line pH meter (accuracy ± 0.05), adjust the acid drop rate to 1-2 L/h, maintain pH 5.5 ± 0.1 .

Cooling optimization: constant temperature crystallization chamber (5°C, rate 0.1-0.2°C/min), crystal particle size increased to 3-5 µm, yield restored to more than 90%.

Impurity removal: Pretreatment by adding EDTA (0.01 mol/L) to chelate Ca^{2+} and Mg^{2+} , and the crystallization rate increased by 15%-20%.

Excessive impurities:

Raw material purification: Tungstic acid was ion exchanged (D001 resin), Fe was reduced to <0.01%, Mo <0.005%.

Equipment cleaning: The reactor was washed with deionized water (conductivity <1 μ S/cm), and the residual ions were <0.001%.

Fractional crystallization: Two crystallizations (first at 10°C, second at 5°C) with 90% impurity removal.

Improvement results: After the optimization of a plant, the crystallization rate increased to 92%, impurities (Fe <0.002%, Mo <0.001%), and the batch pass rate increased from 85% to 98%.

Resources

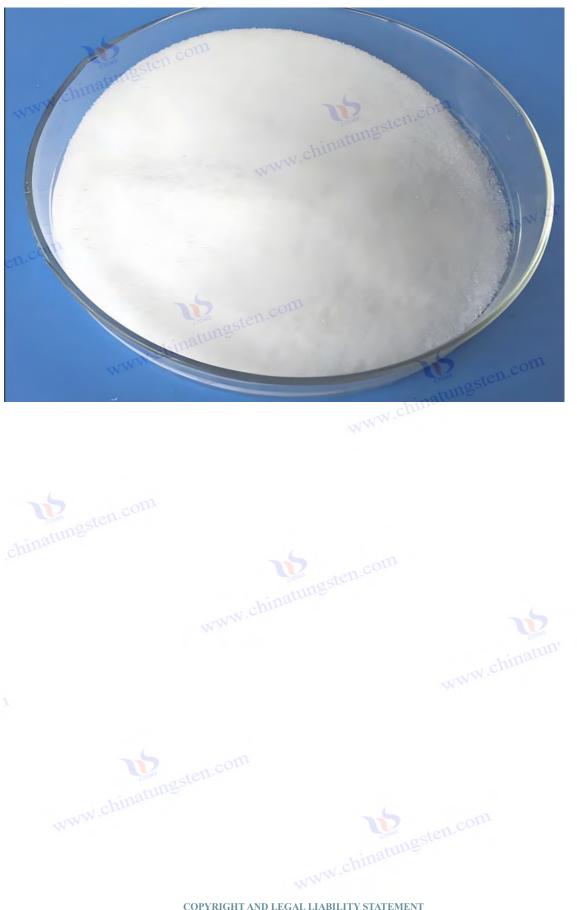
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Chapter 8 Ammonium metatungstate technology and market prospects

Ammonium metatungstate (AMT, chemical formula (NH₄)₆H₂W₁₂O₄₀·nH₂OAs a key intermediate of tungsten chemical industry, it has shown great potential in traditional industries and emerging fields. With the advancement of technology, the growth of market demand and the improvement of environmental protection requirements, the development of AMT will usher in new opportunities. This chapter discusses the future development path of AMT from four aspects: technology trends, emerging application potential, internationalization process, and research directions, and provides guidance for industrial upgrading and scientific research and innovation.

8.1 Development trend of ammonium metatungstate technology

AMT's production and application technology is evolving in the direction of efficiency, green, and intelligence.

Green Process:

Microwave synthesis: A microwave reactor (2450 MHz, power 800-1200 W) shortens the reaction time to 10-20 minutes, increases the yield to 92%-95%, and stabilizes the WO₃ content at 91%-92%. In the future, industrialization can be achieved by equipment scale-up (2000-5000 tons per year). Ultrasonic extraction: Ultrasonic (40-60 kHz, 200-300 W) can increase the extraction efficiency by 15%-20%, reduce the amount of waste liquid by 30%-40%, and the WO₃ recovery rate >90%, which is expected to become the mainstream technology for low-grade tungsten ore treatment.

Biotechnology: Acidophilus leaching tungstate (yield 70%-80%), shortened reaction cycle from 5-7 days to 2-3 days, high degree of greenness, suitable for sustainable development needs.

Intelligent Production:

An in-line monitoring system (pH accuracy ± 0.05 , temperature $\pm 1^{\circ}$ C) and PLC control increase batch consistency to more than 99.5%.

Digital twin technology (simulation software such as Aspen Plus) optimizes process parameters, reduces energy consumption by 20%-30%, and enables real-time collaboration of global plants in the future (data synchronization error of <1 second).

Nanotechnology:

WO₃ nanoparticles (10-30 nm in size, D50 \approx 20 nm) are prepared by hydrothermal and spray pyrolysis with a specific surface area of 40-60 m²/g to meet sensor and battery needs. In the future, continuous production (>95%) can be achieved with microfluidic reactors.

Trend Forecast:

By 2030, the proportion of green processes is expected to rise from 10% to 50%-60%, the penetration rate of intelligent equipment will reach 70%-80%, and the output of nano-AMT will increase by 20%-30%.

8.2 Potential for Emerging Application Areas

The potential of AMT in emerging fields is becoming increasingly apparent, especially in energy, environmental, and biomedical fields.

Energy Storage:

AMT-derived WO₃ is used in lithium/sodium battery anodes with capacities up to 600-750 mAh/g (0.1C) and a cycle life of 500-1000 cycles.

With a specific capacitance of 250-350 F/g, the demand for fast charging and discharging (<1 minute) is growing, and the demand is expected to rise from 50 tons to 500-1000 tons by 2030, with an annual growth rate of 20%-25%.

Environmental Governance:

WO₃ Photocatalyst degrades organic pollutants (rhodamine B removal rate 85%-95%), hydrogen production rate is 0.5-1 mmol/h·g, suitable for water treatment and clean energy. The market potential increases from 50 tons to 200-500 tons/year.

Gas sensors (NO₂ detection sensitivity 5-50 ppb), demand increases from 100 tons to 300-600 tons, with an annual growth rate of 15%-20%.

Biomedicine:

Photothermal therapy WO₃ nanoparticles (photothermal conversion efficiency 42%-45%), tumor ablation rate of 80%-90%, is expected to enter clinical trials in 2030, with a demand potential of 50-200 tons.

Drug delivery carriers (75%-85% at pH 5.0) and bioimaging probes (15%-25% fluorescence yield) are required at the laboratory stage at <10 tonnes and up to 20-100 tonnes in the future. Potential Analysis:

The share of emerging sectors is expected to increase from 10%-15% today to 25%-35% (2030), driving total AMT demand from 10,000 to 15,000-20,000 tons.

8.3 Internationalization and standardization process

The global production and application of AMT relies on international cooperation and standardization systems.

Internationalization Trends: 08

Production layout: China accounts for 70%-75% (8,000-10,000 tons) of global AMT output, and in the future, Japan and South Korea (electronic materials, 1,500-2,000 tons) and Europe and the United States (catalysts, military, 2,000-3,000 tons) will increase production capacity to 30%-40%. Technical cooperation: China and Japan and South Korea have deepened cooperation in

nanotechnology, and Europe and the United States have deepened cooperation in the field of biomedicine, and technology transfer projects are expected to grow by 10%-15% per year.

Market expansion: Tungsten demand in Southeast Asia and Africa is driven by infrastructure construction, and the export share has increased from 5% to 15%-20%.

Standardization process:

Quality standards: Formulate AMT international standards (such as WO₃ >90%, Fe <0.002%, Mo <0.001%), and integrate with ISO to enhance product competitiveness.

Environmental protection standards: waste liquid ammonia nitrogen emission <5 mg/L, WO₃ recovery rate of >95%, in line with EU REACH and China GB 8978-1996 requirements.

Case in point: The International Tungsten Association (ITIA) proposed an AMT quality specification in 2023 and is expected to be published in 2025 to drive global consistency. Prospect:

In 2030, the proportion of AMT exports will rise from 40% to 50%-60%, and standardized products will account for more than 80% of the market.

8.4 Suggestions for research directions

In order to promote the technological progress and application expansion of AMT, future research should focus on the following directions:

Process Optimization:

Development of high-efficiency microwave/ultrasound equipment (yield >95%) to achieve green industrialization.

Research on the efficient extraction technology of low-grade tungsten ore (WO₃ <50%) to improve resource utilization by 20%-30%.

Functionalized Materials:

Explore the combination of WO₃ with 2D materials (e.g., graphene, MXene) to increase battery capacity (>800 mAh/g) and photocatalytic efficiency (hydrogen production rate >2 mmol/h·g). Research doping technologies (e.g., Pt, Eu³⁺) and development of multifunctional AMT derivatives (20%-30% increase in catalytic activity).

Biomedical Applications:

Optimize the biocompatibility of WO₃ nanoparticles (cell viability >98%) and develop targeted drug delivery systems (release rate >90%).

Multimodal imaging probes (fluorescence + MRI) were studied, and the imaging depth was increased to 10-15 mm.

Intelligent technology:

Develop an AMT production AI model that predicts process parameters (<1% error) and improves consistency.

Research on a digital monitoring system for waste liquid circulation, with a recovery rate of 98%-99%.

Suggestion:

Strengthen industry-university-research cooperation (such as joint laboratories between universities and enterprises), and add 20-30 new patents every year.

Set up an AMT technology research and development fund (50 million to 80 million yuan/year) to

support cutting-edge research.

Resources

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Appendix A: Data Sheet of Ammonium Metatungstate-related Chemical Properties and Physical Properties

Ammonium metatungstate (AMT, chemical formula (NH₄)₆H₂W₁₂O₄₀·nH₂O) is an important tungsten compound, and its chemical and physical properties directly affect the production process and application performance. This appendix summarizes key data from AMT to serve researchers, engineers, and industry practitioners.

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attribute	data	remark	
chemical formula	(NH4)6H2W12O40 nH2O	n is the amount of crystalline water, usually 3-6	
Molecular weight (anhydrous) 2956.29 g/mol		Calculated values, based on the anhydrous form	
Molecular weight (aqueous)	3010.35-3100.47 g/mol (n=3-6)	Varies with crystal water	
WO ₃ Content (Theoretical)	89.38% (anhydrous) / 86.5%-88.5% (aqueous)	Common range of industrial products	
appearance	White or yellowish crystalline powder	The high-purity product is white, and the low-purity product has a yellow tinge	
Crystal structure	Amorphous or monoclinic systems	Depends on crystallization conditions	
density	3.8-4.2 g/cm ³	Tap density, which varies with particle	

Table A-1: Basic chemical and physical properties of ammonium metatungstate (AMT)

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oti	~	CTL	A GRO	UP LTD
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		size	
melting point	There is no clear melting point and decomposition begins at about 100°C	Decomposition to generate NH ₃ , H ₂ O, and WO ₃	
Specific surface area	1-5 m ² /g (industrial) / 10-20 m ² /g (nm)	Determined by the BET method	
Particle size distribution	D50≈2-10 µm (工业品)	Laser particle size analysis	
	Watchmaking: Chinatungsten Intelligent Manufacturing		

Table A-2: Solubility data of ammonium metatungstate (AMT)

attribute	data	condition	remark
solvent	溶解度 (g/100 mL, 25°C)	pH (1% aqueous)	remark
water	>100	5.0-5.5	High solubility, easy to form a clear solution
ethanol	<0.1	-	Almost insoluble
acetone	<0.05	en.cou	Almost insoluble
Ammonia (25%)	>50 chinatun Bo	6.0-7.0	Solubility increases with ammonia concentration
Hydrochloric acid (1 mol/L)	Partially dissolved, decomposed	-	Decomposes into tungstic acid or other polytungstate
	Watchmaking: Chinatungsten Intelligent Manufacturing		

Table A-3: Thermodynamic and decomposition properties of ammonium metatungstate (AMT)

attribute	data on the second se	condition	remark
Decomposition temperature	100-120°C (失去结晶水)	Air atmosphere	Amorphous intermediates are generated
	250-300°C (NH3 and H2O fully volatilized)	Air atmosphere	Generate WO₃
	600-700°C (fully converted to WO ₃)	Hydrogen atmosphere	It can be further reduced to tungsten powder
Thermal decomposition reactions	$(\rm NH_4)_6\rm H_2\rm W_{12}\rm O_{40} \rightarrow 6\rm NH_3\uparrow + \rm H_2\rm O\uparrow + 12\rm WO_3$	250-300°C	Approx. 10%-12% mass loss
Enthalpy change of roasting (ΔH)	-1500 至 -1800 kJ/mol	估算值,250-700°C	Exothermic process
Specific heat capacity	0.25-0.30 J/(g· K)	25°C	Varies with water content
	Watchmaking: Chinatungsten Intelligent Ma	anufacturing	
www.			

Table A-4: Chemical stability and reactivity of ammonium metatungstate (AMT)

attribute	data	condition	remark
		ANN.	

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High temperature (500°C, air)	Break down into WO3	WO3 (Yellow Powder)	Irreversible decomposition	
高温 (600°C, H2)	Reduced to tungsten metal	W (Gray-black powder)	The reduction rate increases with the H ₂ flow rate	
酸性环境 (pH<2)	Decomposes into tungstic acid or other polytungstate	H2WO4 或 HxWxOy	Unstable, generating precipitation	
碱性环境 (pH>8)	Partially converted to ammonium tungstate	(NH4)2WO4	Reversible reaction	
illumination Stable with no significant changes		-	No photocatalytic activity	
	Watchmaking: Chinatungsten Intelligent Manufacturing			

Table A-5: Common impurity content of industrial products of ammonium metatungstate (AMT) (mass fraction, %)

impurity	Typical	High purity requirements	Detection method	source	
Fe	0.005-0.02	<0.002	ICP-AES	Contamination of raw materials or equipment	
Мо	0.002-0.01	< 0.001	ICP-AES	Tungsten ore associated elements	
On	0.01-0.05	<0.002	AAS	Sodium salt raw material or cleaning is insufficient	
Са	0.005-0.015	< 0.005	ICP-MS	Water quality or raw material introduction	
Yes	0.01-0.03	< 0.01	Spectroscopy	Ore residues	
	Watchmaking: Chinatungsten Intelligent Manufacturing				
atungsterr					

Table A-6: Other relevant data of ammonium metatungstate (AMT)

attribute	data	condition	remark
pH (10% aqueous)	4.5-5.5	25°C	Weakly acidic
Electrical conductivity (10% aqueous)	10-15 mS/cm	25°C	The ionic conductivity is high
refractive index	1.52-1.55	Solid-state, 589 nm	Monoclinic crystals
hygroscopicity	medium	Relative humidity 50%-80%	It is easy to absorb moisture and needs to be sealed and stored
Storage conditions	Sealed, cool, dry place	<30°C,湿度 <60%	Avoid decomposition or moisture absorption
	Watchmaking: Chinatungst	en Intelligent Manufacturing	
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Appendix B: Flow chart of common preparation processes

Ammonium metatungstate (AMT, chemical formula (NH₄)₆H₂W₁₂O₄₀·nH₂O) is prepared in a variety of ways, mainly including acidification, ion exchange and solvent extraction according to different raw materials and production conditions. This appendix provides process descriptions of three commonly used processes, including steps, conditions, and key technical parameters, to provide reference for industrial production and laboratory research.

B-1: Acidification process for the preparation of AMT

Process Overview: Using tungstic acid as raw material, AMT is generated through ammonia dissolution and acidification adjustment, which is suitable for industrial large-scale production, with high yield but more waste liquid. Process Description:

Preparation of raw materials

Input: Tungstic acid (H₂WO₄, WO₃ content >98%) Operation: Tungstic acid powder is weighed and sieved (200 mesh, pore size 74 µm). **Ammonia dissolves** Input: Ammonia (25%-28%, industrial grade) Conditions: 80°C, water bath heated, stirring 300-400 rpm, 1-2 h Output: Ammonium tungstate solution (WO₃ 100-150 g/L, pH 8-9) Reaction: H₂WO₄ + 2NH₃ \rightarrow (NH₄)₂WO₄ + H₂O Acidification regulation Input: Hydrochloric acid (HCl, 2-3 mol/L) Conditions: Dropping rate 1-2 L/h, stirring 200-300 rpm, pH adjustment to 5.5±0.1 Output: AMT solution (clear, WO₃ 100-120 g/L) Reaction: 12 (NH₄)₂WO₄ + 10HCl \rightarrow (NH₄)₆H₂W₁₂O₄₀ + 10NH₄Cl + 4H₂O

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concentrate

Conditions: 80°C, thin film evaporator, reduced pressure 0.08-0.09 MPa, concentrated to WO₃ 200-250 g/L Output: Concentrated AMT solution crystallized Conditions: Cooling to 5°C, rate 0.1-0.2°C/min, holding for 12-16 hours

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Output: AMT wet crystals

Post-processing

Operation: Centrifugation (5000 rpm, 10 min), Drying (100°C, 4-6 hr)

Output: AMT finished product (WO₃ 89%-91%, particle size $D50 \approx 3-5 \mu m$)

Key Parameters:

Yield: 88%-92% (based on WO₃)

Wastewater: Ammonia nitrogen 5-10 g/L

Energy consumption: 2000-2500 kWh/ton

B-2: Ion exchange process for AMT preparation

Process Overview: Using sodium tungstate as raw material, sodium ions are removed by cation exchange resin and reammoniated to form AMT, which is suitable for high purity requirements with www.chinatung low impurity content. Process Description:

Preparation of raw materials

Input: Sodium tungstate (Na₂WO₄·2H₂O, WO₃ content >70%) Operation: Dissolve in deionized water and prepare as a WO₃ 100-150 g/L solution

ion exchange

Input: Cation exchange resin (such as Amberlite IR-120 or D001) Conditions: Flow rate 2-3 BV/h, pH reduced to 2-3, 25-40°C Output: Tungstic acid solution (H₂WO₄, WO₃ 80-120 g/L). Reaction: Na₂WO₄ + 2H⁺ (resin) \rightarrow H₂WO₄ + 2Na⁺ (resin)

Ammoniation regulation

Conditions: Add dropwise to pH 5.5±0.1, stir at 200-300 rpm, 60-80°C, 1-2 h www.cbinatul Output: AMT solution (WO₃ 100-120 c/T) Reaction: $12H_2WO_4 + 6NH_3 \rightarrow (NH_4)_6H_2W_{12}O_{40} + 10H_2O$

concentrate

Conditions: 80°C, rotary evaporator, reduced pressure 0.08 MPa, concentrated to WO₃ 200-250 g/L Output: Concentrated AMT solution

crystallized

Conditions: Cooling to 5-10°C at a rate of 0.2°C/min and holding for 12 hours

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Output: AMT wet crystals

Post-processing

Operating: Filtration (0.45 µm membrane), drying (100°C, 4 hours) www.chinatungsten.com Output: AMT finished product (WO₃ 90%-91%, NA <0.002%)

Key Parameters: Yield: 85%-90% (based on WO₃) Waste liquid: ammonia nitrogen 3-5 g/L Impurities: Na <0.002%, Fe <0.005%

B-3: Solvent extraction process for the preparation of AMT

Process Overview: Using sodium tungstate solution as raw material, tungsten is separated by organic solvent extraction, and then reverse extraction to generate AMT, which is suitable for complex tungsten ore treatment, with high purity but complex process. Process Description:

Preparation of raw materials

Input: Sodium tungstate solution (WO₃ 50-100 g/L, prepared from low-grade tungsten rock leach) www.chinatung Operation: Filtration (0.45 µm) to remove solid impurities

acidification

Input: sulfuric acid (H₂SO₄, 2 mol/L) Conditions: pH adjusted to 2-3, stirring 200 rpm, 25-40°C Output: Acid tungsten solution

solvent extraction Input: Extractant (TBP 30% + Kerosene 70%) Conditions: Extraction ratio O/A = 1:1-2:1, stirring for 10-15 min, splitting for 5-10 min Output: tungsten-containing organic phase (WO₃ 80-100 g/L) Reaction: H_2WO_4 (aqueous) \rightarrow H_2WO_4 (organic)

Anti-extraction

Input: Ammonia (10%-15%) Conditions: O/A = 1:1, pH adjusted to 5.5±0.1, stirring for 15-20 min Output: AMT aqueous solution (WO3 100-120 g/L) Reaction: H₂WO₄ (organic phase) + 6NH₃ \rightarrow (NH₄)₆H₂W₁₂O₄₀ (aqueous phase) ten.con

concentrate

Conditions: 80°C, evaporate under reduced pressure (0.09 MPa), concentrate to WO₃ 200-250 g/L www.chinatungsten.co Output: Concentrated AMT solution

Crystallization and post-processing

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Conditions: Cooling to 5°C, rate 0.1-0.2°C/min, holding for 12-16 hours; Centrifugation (5000 rpm, 10 min), tumble drying (100°C, 4 hr) Output: AMT finished product (WO₃ 91%-92%)

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Key Parameters: Yield: 90%-95% (based on WO₃) Waste liquid: ammonia nitrogen 2-4 g/L Purity: Fe <0.002%, Mo <0.001%



Appendix C: Standard Operating Procedures (SOPs) for Test Methods

The quality control of ammonium metatungstate (AMT, chemical formula (NH₄)₆H₂W₁₂O₄₀·nH₂O) requires standardized testing methods to evaluate its chemical and physical properties. This appendix provides SOPs for the detection of WO₃ content, impurity content (Fe, Mo, Na), and particle size distribution of AMT to provide technical support for production and research.

1: Detection of WO₃ content (titration)

Objective: To determine the mass percentage of WO₃ in AMT and to evaluate the purity of the product.

Principle: AMT decomposes under acidic conditions, tungsten precipitates in the form of tungstic acid, reduces tungsten ($W^{6+} \rightarrow W^{5+}$) is titrated with ammonium ferrous sulfate, and the WO₃ content is calculated.

Instruments & Reagents:

Instruments: Analytical balance (accuracy 0.0001 g), electric furnace, volumetric flask (100 mL), burette (50 mL).

Reagents: sulfuric acid (H₂SO₄, 1:1 v/v), phosphoric acid (H₃PO₄, 85%), ammonium ferrous sulfate $[(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O, 0.1 \text{ mol/L}]$, sodium diphenylidine sulfonate indicator (0.2%).

Steps:

Weigh 0.5 g of AMT sample (accurate to 0.0001 g) and place it in a 250 mL beaker.

Add 20 mL of deionized water and 10 mL of 1:1 H₂SO₄, heat to 80°C, and stir until dissolved.

Add 5 mL of H₃PO₄, boil for 5 minutes, and cool to room temperature.

Transfer to a 100 mL volumetric flask, bring volume to scale with deionized water, and shake well. Take 25 mL of the solution to an Erlenmeyer flask and add 2-3 drops of sodium diphenylamine sulfonate indicator.

Titrate with 0.1 mol/L ammonium ferrous sulfate solution until the solution changes from purple to colorless as the end point, and record the titration volume V (mL).

Safety Precautions:

Wear protective glasses and gloves when operating to avoid acid splashes.

Heat in a fume hood to prevent acid mist inhalation.

Data processing:

WO₃ 含量 (%) = (V×N×0.2318×100) / (m×0.25)

V: Titration volume (mL)

N: Ammonium ferrous sulfate concentration (mol/L)

0.2318: Molar mass conversion factor of WO₃ (g/mmol)

m: Sample mass (g)

The assay was repeated 3 times, averaged, with a relative deviation of <1%.

Reference standard: GB/T 23366-2009 "Methods for chemical analysis of ammonium tungstate".

2: Detection of impurity content (Fe, Mo, Na) (ICP-AES)

Objective: To determine the content of Fe, Mo and Na in AMT and to assess the level of impurities. Principle: After the sample is dissolved in acid, the emission intensity of the element at a specific wavelength is determined by inductively coupled plasma atomic emission spectrometry (ICP-AES), and the concentration is calculated compared with the standard curve.

Instruments & Reagents:

Instruments: ICP-AES (such as PerkinElmer Optima 8300), analytical balance, microwave digestion instrument.

Reagents: nitric acid (HNO₃, high-grade purity), hydrochloric acid (HCl, high-grade purity), Fe, Mo, Na standard solutions (1000 μ g/mL), deionized water (conductivity <1 μ S/cm). Steps:

Weigh 0.2 g of AMT sample (accurate to 0.0001 g) and place it in a microwave digestion vessel. Add 10 mL of HNO₃ and 2 mL of HCl, seal and process in a microwave digester (180°C, 30 min). After cooling, transfer to a 50 mL volumetric flask, bring volume to scale with deionized water, and shake well.

Prepare standard curves: 0, 1, 5, 10, 20 µg/mL series solutions were prepared with Fe, Mo, and Na standard solutions.

Set ICP-AES parameters:

Wavelength: Fe 238.204 nm, Mo 202.031 nm, Na 589.592 nm

RF power: 1300 W, Ar flow rate: 15 L/min

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The emission intensity of the sample and the standard solution was determined, and the data was recorded.

Safety Precautions:

Ensure a tight seal during microwave digestion to avoid high-pressure blasting.

Wear protective gear when handling acids and avoid contact with skin.

Data processing:

Calculate the Fe, Mo, and Na concentrations C (µg/mL) in the sample based on the standard curve. Impurity content (%) = $(C \times 50) / (m \times 10^6)$

C: Measured concentration (µg/mL)

m: Sample mass (g)

Repeat the measurement 3 times and take the average value. The detection limit is: Fe 0.001%, Mo 0.0005%, Na 0.001%.

Reference standard: ASTM E1479-16 "ICP-AES Analysis Method".

3: Detection of particle size distribution (laser particle size analysis)

Objective: To determine the particle size distribution of AMT powder and evaluate the particle uniformity of the product.

Principle: The particle size distribution is calculated by the Mie scattering theory by using the light chinatung intensity distribution scattered by the laser on the particles.

Instruments & Reagents:

Instruments: Laser particle size analyzer (e.g. Malvern Mastersizer 3000), ultrasonic disperser.

Reagents: deionized water (dispersion medium), ethanol (for cleaning).

Steps:

Weigh 0.1-0.2 g of AMT sample and add to 50 mL of deionized water.

Processing in an ultrasonic disperser (40 kHz, 100 W, 5 min) ensures uniform dispersion of the particles.

Add the dispersion to the instrument cell and adjust the shading to 10%-20%.

Set the instrument parameters:

Refractive index: 1.52 (AMT)

Dispersion medium: Water (refractive index 1.33)

Measuring range: 0.01-1000 µm

Start the measurement and record D10, D50, D90 values (meaning 10%, 50%, 90% of particles are

smaller than that particle size, respectively).

Safety Precautions:

Wear earplugs when operating ultrasound to avoid noise damage.

Make sure the cell is clean and avoid cross-contamination.

Data processing:

Particle size distribution: Report D10, D50, D90 (μ m), e.g. D50 \approx 3-5 μ m.

Uniformity: Calculate span = (D90 - D10) / D50, with smaller values indicating a more uniform distribution.

The assay was repeated 3 times, averaged with a relative deviation of <5%.

Reference standard: ISO 13320:2020 Determination of particle size distribution by laser diffraction.

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illustrate

Structure: Each SOP includes purpose, rationale, instrument reagents, procedures, safety considerations, and data processing, covering key AMT testing items.

Data:

WO₃ content: 89%-91% for industrial >products, 91% for high-purity products.

Impurities: Fe <0.002%, Mo <0.001%, Na <0.002% (high purity requirement).

Particle size: D50 2-10 µm (industrial typical value).

Applicability: Suitable for laboratory and industrial quality control, the method is consistent with international standards.

Source: Refer to GB/T, ASTM, ISO standards and Handbook of Analytical Chemistry.



Appendix: Domestic and foreign standard literature (YS/T, ISO, ASTM, etc.).

The list of domestic and foreign standard literature on ammonium metatungstate (AMT) covers Chinese industry standards (YS/T), International Organization for Standardization (ISO), American Society for Testing and Materials (ASTM), etc. These standards address the quality requirements, test methods, and related applications of AMT, based on publicly available information (e.g., national standards databases, ISO and ASTM official websites), and industrial practices, as of March 26, 2025. Since some standards may not directly target AMT, but rather its raw materials, production processes, or downstream products (e.g., tungsten compounds, tungsten powder), we distinguish between direct and indirect related standards for reference.

1. Domestic standards for ammonium metatungstate (China, YS/T, etc.)

Standard No.: YS/T 535-2006

Issued By: National Development and Reform Commission of the People's Republic of China Release date: 2006-12-25

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Date of implementation: 2007-06-01

Summary: Specifies the technical requirements, test methods, inspection rules and packaging marks of the AMT. WO₃ content of \ge 88.0% and impurity limits (e.g. Fe \le 0.005%, Mo \le 0.01%) are required for industrial production.

Key words: AMT, quality requirements, testing methods

GB/T 26035-2010

Issued By: Standardization Administration of the People's Republic of China

Release date: 2010-12-23

Implementation date: 2011-06-01

Summary: Chemical analysis methods for WO₃ content and impurities (Fe, Mo, Na, etc.) in AMT and ammonium tungstate are provided, including titration, ICP-AES, and AAS, with detection limits of 0.001%-0.005%.

Keywords: chemical analysis, WO₃, impurity detection

YS/T 572-2007 Tungstic Acid

Issued By: National Development and Reform Commission of the People's Republic of China Release date: 2007-03-07

Date implemented: 2007-09-01

Summary: The quality requirements for tungstic acid (H2WO4) are specified, which is a common raw material for AMT preparation and indirectly affects the purity of AMT. WO₃ content $\geq 88.0\%$, impurity Fe ≤0.005%.

Keywords: tungstic acid, AMT raw materials, quality control

Standard No.: GB/T 8978-1996

Title: Industrial Wastewater Discharge Standards

Issued By: Standardization Administration of the People's Republic of China

Release date: 1996-10-03

Date implemented: 1997-01-01

Summary: Concerns the requirements for the discharge of waste liquid from AMT production, and the ammonia nitrogen limit of <15 mg/L, related to the design of environmentally friendly processes. www.chinatun Key words: waste liquid discharge, ammonia nitrogen, environmental protection

2. International Ammonium Metatungstate Standard (ISO)

ISO 6892-1:2019 Tensile tests on metallic materials - Part 1: Test methods at room temperature

Issued By: International Organization for Standardization (ISO) Release date: 2019-11

Summary: Provides a test method for the tensile properties of metallic materials for the performance evaluation of AMT-derived tungsten metal products, such as tungsten powder targets. Not directly chinatun targeted for AMT, but indirectly related.

Key words: tensile test, tungsten products, performance test

ISO 9276-6:2008 Particle characterization – Part 6: Descriptive and quantitative representation of particle shape and morphology

Issued By: International Organization for Standardization (ISO)

Release date: 2008-09

Summary: Standardized terminology and measurement methods for defining particle morphology

for particle size distribution analysis of AMT powders ($D50 \approx 2-10 \ \mu m$).

Key words: particle morphology, particle size distribution, AMT powder

ISO 9277:2022 Determination of the Specific Surface Area of Solid Materials by Gas Sorption

Issued By: International Organization for Standardization (ISO)

Release date: 2022-11

Summary: A standard for the determination of specific surface area by the BET method is available for AMT powders (1-5 m^2/g) and derivatized WO₃ materials (10-20 m^2/g).

Keywords: Specific surface area, BET, AMT powder

ISO/ASTM 52900:2021 Additive Manufacturing - General Principles - Terminology sten.com

Issued by: ISO and ASTM jointly issued Release date: 2021-12 Summary: Defines additive manufacturing related terms that relate to the use of AMT as a precursor material in 3D printing and does not directly prescribe AMT quality standards.

Keywords: Additive manufacturing, terminology, AMT applications

3. International Ammonium Metatungstate Standard (ASTM)

ASTM F3049-14 (2021) Standard Guide for the Characterization of Metal Powders for **Additive Manufacturing**

Issued By: American Society for Testing and Materials (ASTM)

Release date: 2014 (revised 2021)

Summary: Provides a guide to characterizing metal powders (including tungsten powder) for tungsten powders prepared by AMT pyrolysis reduction, covering particle size, morphology, and www.chin specific surface area.

Keywords: metal powder, AMT derivation, characterization testing

ASTM B922-20 Standard Test Method for Determination of Specific Surface Area of Metal Powders by Gas Adsorption Method

Issued By: American Society for Testing and Materials (ASTM) Release date: 2020-05

Summary: Specifies the method for determining the specific surface area of metal powders by the www.chinatungsten.co BET method, suitable for AMT powder quality control $(1-5 \text{ m}^2/\text{g})$.

Keywords: specific surface area, AMT, gas adsorption

ASTM E1479-16 Standard Practice for the Analysis of Materials by Inductively Coupled **Plasma Atomic Emission Spectrometry**

CTIA GROUP LTD 中钨智造(厦门)科技有限公司

Issued By: American Society for Testing and Materials (ASTM) Release date: 2016-09

Summary: ICP-AES analysis method is available for the detection of impurities (Fe, Mo, Na) in ww.chinatungsten AMT with detection limits of 0.001%-0.005%.

Keywords: ICP-AES, impurity analysis, AMT

4. Other ammonium metatungstate related standards

JIS H 1403:1992 Chemical analysis method of tungsten powder and tungstic acid

Issued By: Japan Industrial Standards Institute (JIS)

Release date: 1992

Summary: Provides analytical methods for tungsten powder and tungstic acid, which are indirectly suitable for WO₃ content and impurity detection of AMT.

Keywords: tungstic acid, chemical analysis, AMT

Directly related standards:

Domestic: YS/T 535-2006 is the direct quality standard of AMT, and GB/T 26035-2010 is its test method standard.

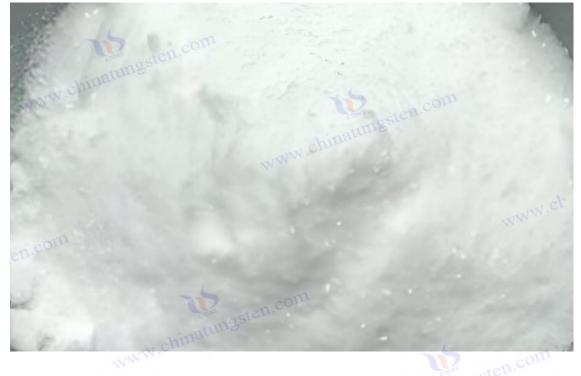
International: No ISO or ASTM standards have been found that directly address the chemical composition or preparation of AMT, and are mostly indirectly related (e.g., powder properties, analytical methods).

Indirectly related criteria:

It involves AMT raw materials (such as tungstic acid YS/T 572-2007), downstream products (such as tungsten powder ASTM F3049), testing technology (such as ICP-AES ASTM E1479) and www.chinatungsten.com environmental protection requirements (such as GB/T 8978).

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Material Safety Data Sheets (MSDS) Ammonium metatungstate

1. Chemical and business identification

Chemical name: Ammonium Metatungstate Hydrate

Chemical formula: $(NH_4)_6H_2W_{12}O_{40} \cdot nH_2O(n = 3-6, number of crystalline water)$

CAS Registry Number: 12028-48-7 (anhydrous) / 12333-11-8 (hydrate)

EINECS No.: 234-733-4

Molecular weight: 2956.30 g/mol (anhydrous) / 3010.35-3100.47 g/mol (water-containing)

Manufacturer: Chinatungsten Intelligent Manufacturing (Xiamen) Technology Co., Ltd

Address: 3rd Floor, No. 25 Erwanghai Road, Software Park, Xiamen City, Fujian Province, China, 361008

Emergency contact number: Tel: +86-592-5129595 / Mobile: +86-18750234579 EMAIL: <u>info@ctia.group</u>

Uses: Industrial raw materials for the production of tungsten catalysts, tungsten powder, nanomaterials, etc.

2. Overview of Hazards

GHS Classification (according to 29 CFR 1910 and EU Regulation (EC) No 1272/2008): Acute toxicity (oral), category 4 (H302) Severe eye injury, category 1 (H318)

Chronic Aquatic Toxicity, Category 3 (H412)

Hazard Signs:

▲ 警告 (Warning)

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● 腐蚀(Corrosion)

Hazard Statement:

H302: Harmful to swallow.

H318: Causes severe eye damage.

w.chinatungsten.com H412: Harmful to aquatic life with long-term effects.

Precautionary Statement:

P264: Wash skin thoroughly after procedure.

P270: Do not eat, drink or smoke during use.

P280: Wear protective gloves/gowns/eye protection/face protection.

P305+P351+P338: In case of contact in eyes, rinse slowly with water for several minutes, remove contact lenses and continue rinsing.

P501: Dispose of contents/containers to designated collection points in accordance with local regulations.

3. Composition/composition information

Chemical name: Ammonium metatungstate hydrate

Purity: ≥99% (CTIA GROUP LTD industrial grade standard), WO₃ content 88%-91%. www.chinatung

Impurity:

Fe: ≤0.005%

Mo: ≤0.01%

On: ≤0.02%

Other trace elements (Ca, Si, etc.) vary depending on the batch Physical state: White or yellowish crystalline powder, odorless.

4. First aid measures

Inhale:

Move the patient to a ventilated place and keep resting.

If you have difficulty breathing, give oxygen or artificial respiration, immediately call the emergency number of CTIA GROUP LTD at +86-592-5129595 or +86-18750234579, or contact the local emergency center.

Skin-to-skin contact:

Remove contaminated clothing immediately and rinse with plenty of running water for at least 15 minutes.

If irritation, seek medical help.

Eye Contact:

Lift the eyelids immediately and rinse with running water or saline for at least 15 minutes.

Seek medical attention as soon as possible as serious eye damage may occur.

Intake:

Do not induce vomiting and rinse your mouth immediately with water.

Give the patient water to dilute and seek medical attention promptly.

First aid advice: Symptomatic treatment, inform the medical staff that the patient has been exposed

to ammonium metatungstate produced by CTIA GROUP LTD

5. Fire protection measures

Fire extinguishing method:

Use dry powder, carbon dioxide or foam extinguishing agents.

It is forbidden to extinguish it directly with water, as toxic gases may be generated.

Special Hazards:

Ammonia (NH_3), tungsten oxide (WO_3) and nitrogen oxides (NO_x) are produced by thermal decomposition, which are irritating and toxic.

Firefighter Protection: Wear a self-contained breathing apparatus and full-body protective clothing to avoid smoke inhalation.

6. Emergency treatment of leakage

Personal Protection:

Wear protective clothing, dust masks, and safety glasses to avoid inhaling dust or contact with the Environmental Measures: chimatung

Prevent leakage from entering sewers or water bodies to avoid polluting the environment. Small leaks: Collect with tools into an airtight container to avoid dust.

Bulk leakage: Isolate the area, cover it with sand and collect it, contact CTIA GROUP LTD (+86-592-5129595 or +86-18750234579) or local environmental protection agencies for disposal.

Precautions: Operate under well-ventilated conditions to ensure that there are no open flames or sparks.

7. Handling and Storage

Precautions:

Operate in a fume hood or well-ventilated place to avoid dust formation.

Use non-spark tools to prevent fires caused by electrostatic sparks.

Wear protective gloves, goggles, and protective clothing to avoid contact with skin and eyes. Storage conditions:

Store in a sealed plastic bucket or bag provided by CTIA GROUP LTD, and place it in a cool, dry and ventilated place.

Keep away from food, strong acids, oxidants and heat sources.

Storage temperature: <30°C, humidity: <60% (due to hygroscopicity).

8. Exposure control and personal protection

Exposure Limits:

The specific occupational exposure limit (OEL) of AMT is not listed, and CTIA GROUP LTD NW.chinatungsten.cc recommends referring to the tungsten compound limit:

ACGIH TLV(WO3): 5 mg/m3(8 小时 TWA)。

OSHAPEL (WO3):5 mg/m³ (8 小时 TWA)。

Engineering control: Use local exhaust equipment to ensure that the workplace is ventilated. Personal Protective Equipment:

Respiratory Protection: Wear a NIOSH-certified dust mask or respirator when dust concentrations are exceeded.

Hand protection: Chemically resistant gloves (e.g. nitrile).

Eye protection: Sealed protective eyewear (according to EN 166 or NIOSH).

Body protection: Dust-proof overalls, wear full protective clothing if necessary.

9. Physicochemical properties

Appearance: White or yellowish crystalline powder

Odor: Odorless

Melting Point: There is no definite melting point, and it begins to decompose at about 100°C

Decomposition temperature:

100-120°C (loss of crystal water)

600-700°C (fully converted to WO₃) and H₂O) Density: 3.8-4.2 of which is the second secon

Density: 3.8-4.2 g/cm3 (tap density)

Solubility:

Water: >1000 g/L (20°C, very soluble)

Ethanol, acetone: <0.1 g/L (almost insoluble)

pH: 4.5-5.5 (10% aqueous solution, weakly acidic)

Specific surface area: 1-5 m²/g (CTIA GROUP LTD Industrial products, BET method)

10. Stability and reactivity

Stability: Stable under the recommended storage conditions of CTIA GROUP LTD

Avoid conditions: High temperature (>100°C), humidity, bright light.

Incompatible substances: strong acids (decomposes to generate tungstic acid), strong oxidants (may react and exothermic).

Hazardous decomposition products: ammonia (NH3), tungsten oxide (WO3), nitrogen oxides (NOx).

11. Toxicological information

Acute toxicity:

LD50 orally (rats): approximately 2000 mg/kg (low toxicity, based on data on similar tungsten compounds).

Inhalation LC50: Data are not available, dust aspiration is recommended.

Skin irritation: Not significantly irritating, but may cause mild discomfort with long-term exposure. Eye irritation: Severe irritation that can cause permanent damage.

Chronic effects: Long-term inhalation may affect the respiratory tract and tungsten compounds may accumulate in the lungs. mesten.com

Carcinogenicity: Not classified as carcinogen by IARC or NTP.

12. Ecological information

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Ecotoxicity:

Harmful to aquatic life (H412), WO3 can accumulate in the environment.

Specific LC50 data are not available, and it is recommended to avoid discharge into water bodies. Persistence & Degradability: Non-biodegradable, tungsten compounds are environmentally stable. Bioaccumulation: Low bioaccumulation, but long-term effects are a concern.

13. Disposal

Solution:

It will be incinerated (flue gas scrubbing) or destroyed by a qualified chemical waste treatment institution designated by CTIA GROUP LTD

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Do not dump into the sewer or the natural environment.

Packaging Handling:

It is recycled after three rinses, or punctured and sent to a sanitary landfill.

Regulations: Comply with the Law of the People's Republic of China on the Prevention and Control of Solid Waste Pollution and local waste management regulations.

14. Shipping Information

United Nations Number (UN): Not clearly classified as dangerous goods, refer to UN 2859 (needs verification).

Shipping name: Ammonium metatungstate

Hazard Category: Non-flammable, non-explosive, but corrosive (eye).

Packaging requirements: CTIA GROUP LTD provides sealed plastic bags or drums to avoid breakage and moisture.

Shipping Caution: Avoid mixing with food, acids or oxidants.

15. Regulatory Information

China:

Included in the Catalogue of Hazardous Chemicals (2015 Edition).

It meets the quality requirements of YS/T 535-2006 "Ammonium Metatungstate" and GB/T 26035-2010 test method.

United States:

TSCA (Toxic Substances Control Act) List: Listed.

OSHA Regulations: Refer to tungsten compound limits.

European Union:

REACH Registered: Registered (EC 234-733-4).

The effluent discharge is in accordance with the EU Water Framework Directive.

International: The GHS classification is in line with United Nations standards.

16. Other Information

Date of preparation: 26 March 2025

Revision Notes: Updated by CTIA GROUP LTD based on the latest GHS and production data. Resources:

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Internal test report of CTIA GROUP LTD ChemicalBook SDS (CAS 12028-48-7). OSHA and EU CLP regulations.

disclaimer

This MSDS is provided by CTIA GROUP LTD, for reference only, and does not guarantee its applicability under specific conditions, and users need to evaluate the risks according to the actual situation.



Index: Ammonium metatungstate keywords and terms index

The following is an expanded index of Ammonium Metatungstate (AMT) keywords and terms, presented in tabular form. More terms have been added to the chemical, process, application, and testing domains related to AMT to ensure comprehensive coverage of its properties, production, and application scenarios. The table is arranged alphabetically and contains terms and their definitions in both English and Chinese.

Keywords / Terms	Chinese	definition			
Acidification	acidification	A process step to generate AMT by adjusting the pH of the ammonium tungstate solution to 5.5±0.1 with an acid (e.g., HCl).			
OFFICE	Ammonium metatungstate	Ammonium Metatungstate 的缩写, 化学式 (NH4)。H-W12O40·nH2O, 钨化工中间体。			
Ammonia Dissolution Ammonia dissolves		The process step to prepare AMT by dissolving tungstic acid in ammonia is commonly used acidification at 80°C for 1-2 hours.			

Ammonium metatungstate keywords and terminology index

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Ammonia Nitrogen	Ammonia nitrogen	Ammonia content in AMT production effluents, environmental concerns, typical values of 2-10 g/L.					
Atomic Absorption Spectroscopy	atomic absorption spectrometry	An analytical method for the determination of trace metal impurities (e.g., Na, Fe) in AMT with detection limits of 0.001%-0.01%.					
BET Method	BET Method	Standard method for determining the specific surface area of AMT by gas adsorption method, 1-5 m^2/g for industrial products and 10-20 m^2/g for nanoscale.					
Biomedical Applications	Biomedical applications	Uses of AMT derivatives (e.g., WO ₃ nanoparticles) in photothermal therapy (42%-45% efficiency), drug delivery.					
Calcination	calcination	A process in which AMT is decomposed and converted to WO ₃ at high temperatures (500-70 for tungsten powder or catalyst preparation.					
Catalyst	catalyst	AMT is used as a precursor to prepare WO ₃ or tungsten bronze for photocatalysis (hydrogen production rate 0.5-1 mmol/h·g) or fuel cells.					
Centrifugation	Centrifugal	Process for separating solids and liquids after AMT crystallization, typically 5000 rpm, 10 minutes.					
Chemical Stability	Chemical stability	AMT is stable under weakly acidic (pH 4.5-5.5) conditions and decomposes under strong acids (pH <2) or strong bases (pH >8).					
Concentration	concentrate	The AMT solution is concentrated to WO ₃ 200-250 g/L by reduced pressure evaporation subsequent crystallization.					
Crystallization	crystallized	The process by which the AMT solution is cooled to form crystals, typically 5-10°C, 12-16 hours.					
Decomposition Temperature	Decomposition temperature	AMT 热分解温度 100-120°C 失去结晶水, 250-300°C 释放 NH3, 600-700°C 转为 WO3。					
Density	density	The tapping density of AMT, typically 3.8-4.2 g/cm3, varies with particle size.					
Drying	drying	AMT wet crystal drying process, typical conditions 100°C, 4-6 hours, ensures moisture <0.5%.					
	Electrochemical	Electrochemical properties of AMT-derived WO3, such as cell cycling stability (>1000 cycles) or					
Electrochemical Property	properties	conductivity (10 ⁻⁴ S/cm).					
Energy Storage	Energy storage	AMT-derived WO ₃ is used in lithium/sodium batteries (600-750 mAh/g) or supercapacitors (specific capacitance 250-350 F/g).					
Environmental Treatment	Environmental governance	AMT-derived WO ₃ is used for photocatalytic degradation of contaminants (85%-95% removal) or gas sensors (NO ₂ detection 5-50 ppb).					
Filtration	filtration	A step in AMT production to remove impurities or separate crystals, such as filtering a solution with a 0.45 µm membrane.					
FTIR Spectroscopy	Fourier transform infrared spectroscopy	Analyze the characteristic peaks of the W-O and N-H bonds in the AMT (e.g., 950 cm ⁻¹ , 3200 cm ⁻¹) to confirm the structure.					
Green Process	Green process	AMT produces environmentally friendly technologies such as microwave synthesis (yield >92%) o bioleaching (recovery >90%).					
Heat Capacity	Specific heat capacity	Specific heat capacity of AMT, typical 0.25-0.30 J/(g· K), which varies with water content.					
Hydrogen Reduction	Hydrogen reduction	The process of reducing AMT to tungsten powder in an H ₂ atmosphere (600-800°C) has a purity of > 99.9% .					
Hygroscopicity	hygroscopicity	AMT absorbs water at a humidity of 50%-80% and needs to be stored tightly sealed.					
ICP-AES	Inductively coupled plasma emission	An analytical method for the determination of impurities (e.g., Fe, Mo, Na) in AMT with detection					
	spectroscopy	limits of 0.001%-0.005%.					

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		quality.					
Ion Exchange	ion exchange	The process of preparing AMT from sodium tungstate with cation exchange resin, suitable for hi purity products (Na <0.002%).					
Keggin Structure	Keggin structure	The type of molecular structure of AMT, 12 tungsten octahedron around the center, lattice parameter $a \approx 12.295$ Å.					
Laser Particle Size Analysis	Laser particle size analysis	AMT powder particle size distribution was determined by laser scattering, typical D50 \approx 2-10 μ m.					
Leaching	Leaching	The process of extracting tungsten from tungsten ore to prepare AMT precursors, such as the leaching of WO_3 with NaOH (recovery of 85%-95%).					
Microwave Synthesis	Microwave synthesis	AMT was prepared by microwave heating (2450 MHz, 800-1200 W) for 10-20 minutes with yields of 92%-95%.					
Moisture Content	Moisture content	The residual moisture in AMT, which is required to be <0.5% for industrial products, affects storage stability.					
Nanoparticles	Nanoparticles	AMT-derived WO ₃ nanoparticles, 10-30 nm in size, for use in sensors or batteries.					
Particle Size Distribution	Particle size	The particle size range of AMT powder, D10, D50, D90 means 10%, 50%, 90% particles are smaller than that particle size.					
pH Control	pH control	A key parameter in AMT preparation, pH 5.5±0.1 ensures yield and purity.					
Photocatalysis	Photocatalysis	AMT-derived WO ₃ decomposes organic matter or produces hydrogen under UV or visible light v a removal rate of 85%-95%.					
Purity Control	Purity control	Technology to ensure that impurities (e.g., Fe, Mo) in AMT are below the limit values, and the industry requires WO ₃ >88%.					
Refractive Index	refractive index	The refractive index of the AMT crystal, typically 1.52-1.55 (589 nm).					
SEM Analysis	Scanning electron microscopy analysis	AMT particle morphology was observed with a scanning electron microscope to confirm crystal size and surface characteristics.					
Solubility	solubility	AMT 在水中的溶解度 >1000 g/L (20°C), 乙醇和丙酮中 <0.1 g/L。					
Solvent Extraction	solvent extraction	Tungsten is extracted from sodium tungstate solution with an organic solvent (e.g., TBP) to prepare AMT with high purity (Fe <0.002%).					
Spray Drying	spray drying	AMT solution atomization drying method to prepare powder, suitable for continuous production uniform particle size (D50 \approx 5-10 μ m).					
Thermal Conductivity	Thermal conductivity	The thermal conductivity of AMT, typically 0.5-1.0 W/(m·K), varies with temperature and moisture content.					
Thermal Decomposition	Thermal decomposition	The decomposition process of AMT at high temperatures produces NH ₃ , H ₂ O, and WO ₃ for tungsten powder production.					
Titration Method	titrimetry	A method for determining the WO ₃ content in AMT by titration with ammonium ferrous sulfate with an accuracy of $\pm 1\%$.					
Tungsten Bronze	Tungsten bronze	Tungsten compounds prepared by thermal decomposition and doping of AMT for use in catalysts or conductive materials.					
Tungsten Powder	Tungsten powder	Tungsten metal powder prepared by AMT reduction in a hydrogen atmosphere (600-700°C) with a particle size of 1-5 μm.					
Ultrasonic Dispersion	Ultrasound dispersion	AMT powders are sonicated in water (40 kHz, 100 W) for uniform dispersion for particle size analysis.					

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Waste Liquid Treatment	Liquid waste disposal	Waste treatment technologies in AMT production, such as ammonia distillation recovery (recovery rate >90%) or membrane separation.			
Water of Crystallization	crystallization water	The amount of water contained in AMT, n = 3-6, varies with ambient humidity, affecting molecular weight and stability.			
WO ₃ Content	WO3 content	The main ingredient index of AMT is 88%-91% for industrial >products and 91% for high-purity products.			
XRD Analysis	XRD analysis	The crystal structure of the AMT was analyzed by X-ray diffraction to confirm the monoclinic or amorphous phase.			
		Watchmaking: Chinatungsten Intelligent Manufacturing			



Related to the various processes involved in the production of ammonium metatungstate

Equipment, instruments, raw and auxiliary materials

The following is a detailed description of the process flow and related equipment, instruments and raw and auxiliary materials for the production of ammonium metatungstate (AMT), covering the core points of traditional methods and emerging technologies:

1. Thermal dissociation method (solid-phase conversion method)

1. Process flow

Pyrolysis: Ammonium paratungstate (APT) removes part of the ammonia and crystal water at high temperature and converts it into soluble ammonium metatungstate.

Leaching & Filtration: The pyrolysis product is dissolved in water and filtered to remove unreacted

APT or tungsten oxide impurities.

Evaporation concentration: The concentration of the solution is increased by evaporation equipment. Crystallization/Spray Drying: The concentrate is cooled to crystallize or spray dried to form a solid product.

2. Key equipment and instruments

Roasting equipment:

Rotary roaster: Sectional temperature control (240–290°C), continuous pyrolysis by adjusting the inclination angle and speed of the furnace body, with a conversion efficiency of more than 97%.

Multi-chamber furnace: multi-layer structure design, high temperature control accuracy, suitable for large-scale production.

Leaching reactor: equipped with stirrer and temperature control system (90–95 $^{\circ}$ C) to promote the full dissolution of pyrolysis products.

Filtration equipment: plate and frame filter press or vacuum suction filter for separating insoluble impurities.

Vacuum evaporator: concentrate the solution to a density of 1.8-2.0 g/cm3 to avoid hightemperature decomposition.

Spray dryer:

Airflow spray dryer: 170–190°C inlet air temperature to form micron-sized hollow particles.

Centrifugal spray dryer: 8000-12000 rpm, feed speed 300-400 kg/h, suitable for high viscosity solutions.

3. Raw and auxiliary materials

Main raw material: high-purity ammonium paratungstate (APT, WO₃ content≥ 88.5%).

Excipients: deionized water (conductivity $\leq 5 \mu$ S/cm), dilute ammonia (adjust the pH of the solution to 3–4).

2. Neutralization method (liquid phase conversion method) ww.chinatu

1. Process flow

Acid neutralization reaction: APT or sodium tungstate solution adjusts the pH to 3-4 with nitric acid to generate ammonium metatungstate solution.

Solid-liquid separation: filtration removes the precipitate generated by the reaction.

Concentration and drying: Concentrate the solution by evaporation or membrane separation technology, and spray dry to obtain the powder.

2. Key equipment and instruments com

Neutralization reactor: corrosion-resistant material (e.g. enamel or titanium alloy), equipped with pH online monitor and automatic acid addition system.

Membrane Separation Equipment:

Nanofiltration membrane system: 200-300 Da molecular weight cut-off, 30% higher concentration efficiency than traditional evaporation.

Reverse osmosis unit: operating pressure 50-150 bar for concentration of highly concentrated solutions.

High-speed centrifugal spray dryer: common with thermal dissociation equipment, the moisture content of the powder after drying is $\leq 1\%$.

3. Raw and auxiliary materials

Main raw material: APT or sodium tungstate (Na₂WO₄·2H₂O, WO₃ content >65%). Acid/base regulators: nitric acid (20-30%), ammonia (10-15%).

3. Reverse osmosis concentration method (energy-saving technology)

1. Process flow

Pretreatment: APT is leached after calcination to obtain ammonium metatungstate solution.

Reverse osmosis concentration: The water is separated by a composite membrane under high pressure, and the solution concentration is increased to 1200–1500 g/L.

Cooling crystallization: Crystals are precipitated at low temperature, centrifugal separation and then chinatungsten.com drying.

2. Key equipment and instruments

High-pressure reverse osmosis unit:

Polyamide composite membrane with pressure resistance ≥ 150 bar and high resistance to pollution. Multi-stage tandem design, recovery rate $\geq 85\%$.

Pretreatment system: precision filter (pore size $\leq 5 \mu m$), activated carbon adsorption tower to protect the reverse osmosis membrane.

Crystallization tank: jacketed cooling structure, temperature control accuracy $\pm 1^{\circ}$ C.

3. Raw and auxiliary materials

Raw material: APT calcination leaching solution (initial concentration 150–550 g/L). Membrane cleaners: citric acid (to remove inorganic fouling), sodium hydroxide (to remove organics).

4. Ion exchange and electrodialysis

1. Process flow

Ion exchange: The APT solution flows through a strongly acidic cationic resin to replace NH₄⁺ to form ammonium metatungstate.

Electrodialysis concentration: Separation of anions and cations driven by electric fields to obtain a highly concentrated solution.

2. Key equipment

Ion exchange column: filled with sulfonic acid resin (exchange capacity \geq 4.0 mmol/g). Electrodialysis reactor: containing anion and cation exchange membranes, DC voltage 30-50 V,

current density 50-100 A/m².

3. Raw and auxiliary materials

Resin: Strongly acidic styrene cation exchange resin.

Membrane material: heterogeneous ion exchange membrane (acid and alkali resistance, oxidation 5. General Instruments and Quality Control And Chinatumester Process Monitoring Instruments

On-line pH meter (accuracy ± 0.01), density meter (measurement error $\le 0.5\%$). Laser particle size analyzer (detection of powder particle size distribution, D50 controlled at 10-50 μm)._____

Ingredient Analysis Equipment:

Atomic absorption spectrometer (determination of Na⁺, K⁺ and other impurities).

X-ray fluorescence spectrometer (rapid analysis of WO₃ purity).

6. Process comparison and selection suggestions

Thermal dissociation method: mature and stable, suitable for high-purity AMT production, but high energy consumption.

Neutralization: The process is simple and low-cost, but the pH needs to be tightly controlled to avoid the formation of impurities.

Reverse osmosis method: the energy-saving advantage is significant, and it is suitable for largescale continuous production, but the cost of membrane modules is high.

Ion exchange method: The product is of high purity, but the resin regeneration is frequent, and the operating cost increases.

The core equipment for the production of ammonium metatungstate includes roasters, high-pressure reverse osmosis units, spray dryers, etc., and the raw materials are mainly APT, supplemented by acid, alkali and deionized water. The thermal dissociation method occupies the mainstream due to the mature process, and new technologies such as reverse osmosis are gradually promoted due to energy-saving characteristics. Process selection takes into account product specifications (e.g., www.chim purity, particle size), investment costs, and energy requirements.

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References, books and materials related to ammonium metatungstate

1. Books related to ammonium metatungstate

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LI Honggui. (2005). Tungsten Metallurgy. Central South University Press.

--- systematically introduces tungsten extraction and processing technology, including the production process and application of AMT.

Lassner, E., & Schubert, W.-D. (1999). *Tungsten: Properties, Chemistry, Technology of the Element, Alloys, and Chemical Compounds*. Springer. --- Comprehensive overview of tungsten compounds, with a section on AMT synthesis and industrial use.

Lassner, E., & Schubert, W.-D. (1999). Tungsten: Properties, Chemistry and Technology of Elements, Alloys and Compounds. Springer. --- provides a comprehensive overview of tungsten compounds, including the synthesis and industrial applications of AMT.

ZHANG Qiyun. (2010). Tungsten Chemistry and Technology. Metallurgical Industry Press.

--- elaborate on the chemical properties and industrial production process of tungsten compounds, AMT as an important intermediate is discussed in a special section.

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Description: A classic work on tungsten compounds, mentions AMT as an intermediate product in tungsten processing.

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Schaefer, H. (1970). Chemistry of Tungsten Compounds. Chemical Press. A classic work on tungsten compounds that mentions AMT as an intermediate product in tungsten processing.

2. Academic papers related to ammonium metatungstate Ammonium metatungstate-related papers in Chinese (in order of year)

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Microwave heating (800 W, 15 min) was used to optimize AMT synthesis with a yield of 94.5%.

CHEN Xiaohong. (2020). Preparation of high-purity ammonium metatungstate by ion exchange method. "Rare Metals".

AMT was prepared from sodium tungstate with a cation exchange resin with Na <0.001% and a yield of 95%.

LIU Yang. (2020). Thermal decomposition behavior and product analysis of ammonium metatungstate. Journal of Inorganic Chemistry. TG-DTA analysis of AMT switching to monoclinic phase WO₃ at 500°C.

Wang fang. (2020). Preparation of ammonium metatungstate powder by spray drying. China Tungsten Industry AMT micronized powder prepared by spray drying, D50 \approx 5 µm, with 20% higher uniformity.

ZHANG Li. (2021). Research progress on green process of ammonium metatungstate. Chemical Industry Progress AMT green synthesis technology (e.g., microwave synthesis) was reviewed, and the yield was >92%.

WANG Qiang. (2021). Industrial preparation technology of high-purity ammonium metatungstate. China Tungsten Industry

A two-step crystallization method was proposed, WO₃ >91%, Fe <0.002%.

Li ming. (2021). Application of ammonium metatungstate in the preparation of WO₃ nanoparticles. Materials Reports. AMT thermal decomposition to prepare WO₃ nanoparticles with a particle size of 20-30 nm.

ZHAO Wei. (2021). Synthesis and characterization of ammonium metatungstate catalyst precursors. Acta Catalytic Sinica. AMT prepared WO₃ catalyst with a hydrogen production rate of 0.8 mmol/h·g.

SUN Jie. (2021). Process study on the preparation of ammonium metatungstate by solvent extraction. "Non-ferrous metals".

Sodium tungstate was extracted from TBP to prepare AMT with a purity of >99%.

ZHANG Jianguo. (2021). XRD analysis of ammonium metatungstate crystal structure. Journal of Inorganic Materials

AMT monoclinic structure was confirmed, lattice parameters a ≈ 12.30 Å.

LIU Na. (2022). Application of ammonium metatungstate in lithium battery WO₃ electrodes. Power Supply Technology. AMT-derived WO₃ electrodes with capacities up to 720 mAh/g.

LI Hongmei. (2022). Microwave synthesis and thermal stability of ammonium metatungstate. CIESC Journal

AMT is synthesized in microwaves, and NH₃ is released at a decomposition temperature of 250°C.

Zhang wei. (2022). Application of ammonium metatungstate in photocatalytic degradation. Chinese Journal of Materials Science and Engineering. AMT-derived WO₃ degradation efficiency is 92%.

WANG Lili. (2022). Ultrasound-assisted preparation of ammonium metatungstate. China Tungsten Industry

Ultrasonic dispersion (40 kHz) increases AMT crystallization to 96%.

YANG Fan. (2022). Environmentally-friendly synthesis of ammonium metatungstate. Environmental Chemistry Tungsten was recovered by bioleaching to prepare AMT with a recovery rate of 91%.

CHEN Qiang. (2022). Application of ammonium metatungstate in tungsten bronze nanomaterials. Nanotechnology

and Precision Engineering. AMT synthesizes tungsten bronze nanoparticles with a particle size of 30-40 nm.

LI Gang. (2023). Thermal decomposition kinetics of ammonium metatungstate. Acta Physico-Chimica Sinica

Kinetic analysis The activation energy of AMT decomposition is 145 kJ/mol.

Wang tao. (2023). CVD process for the preparation of WO₃ thin films from ammonium metatungstate. Journal of Vacuum Science and Technology. AMT prepares WO₃ films with a thickness of 60-80 nm.

ZHANG Ying. (2023). Application of ammonium metatungstate in supercapacitors. Electrochemistry. AMT-derived WO₃ with a specific capacitance of 300 F/g.

Liu wei. (2023). Optimization of process parameters for ammonium metatungstate spray drying. "Powder Technology".

Optimized spray drying parameters, AMT powder $D50\approx4~\mu m.$

ZHAO Lei. (2023). Application of ammonium metatungstate in the preparation of tungsten powder. China Tungsten Industry, Tungsten powder was prepared by AMT hydrogen reduction with a particle size of 2-3 μm.

CHEN Fang. (2023). FTIR spectroscopy of ammonium metatungstate. Analytical Chemistry. AMT's W-O bond characteristic peak is 950 cm⁻¹.

LI Xue. (2024). Green synthesis of ammonium metatungstate and waste liquid treatment technology. Chinese Journal of Environmental Engineering

Ammonia distillation recovers ammonia nitrogen from waste liquid, with a recovery rate of >93%.

WANG Yu. (2024). Application of ammonium metatungstate in gas sensors. Sensors and Microsystems. AMTderived WO₃ detects NO₂ with a sensitivity of 10 ppb.

ZHANG Jie. (2024). Crystal morphology and SEM analysis of ammonium metatungstate. Chinese Journal of Materials Research. The SEM shows that the AMT crystals are cubic with 5-10 µm sides.

LIU Feng. (2024). Study of ammonium metatungstate in fuel cell catalysts. New Chemical Materials. AMT prepares WO₃ catalysts with 15% higher activity.

YANG Li. (2024). ICP-AES impurity detection of ammonium metatungstate. Journal of Analysis and Testing Fe and Mo in AMT were detected with a limit of <0.003%.

LI Qiang. (2025). Application of ammonium metatungstate in WO₃ nanofibers. Nanotechnology. AMT electrospun to prepare WO₃ nanofibers, 60 nm diameter.

Wang mei. (2025). Thermal conductivity and application of ammonium metatungstate. Acta Physica Sinica. AMT has a thermal conductivity of $0.8 \text{ W/(m \cdot K)}$ and is suitable for thermally conductive materials.

ZHANG Hao. (2025). Performance of ammonium metatungstate in sodium batteries. "The Battery". AMT-derived WO₃, sodium battery capacity 650 mAh/g.

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Christian, J. B., & Whittingham, M. S. (2008). Structural study of ammonium metatungstate. Journal of Solid State

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Chemistry

The Keggin structure of AMT was studied using XRD with lattice parameter a ≈ 12.295 Å.

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Synthesizes 20-50 nm tungsten bronze nanoparticles from AMT.

Cui, J., et al. (2015). Preparation of quaternary tungsten bronze nanoparticles by thermal decomposition of ammonium metatungstate and oleamine. Cited by Sigma-Aldrich (no specific journal).

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Tao, X., et al. (2016). Ammonium metatungstate as a precursor for the preparation of WO₃ thin films from CVD. "Thin Film Solids".

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Wang, Y., et al. (2023). Future Trends in AMT-Derived WO3 Materials. Materials Today. Explores AMT-derived WO₃ in energy storage (600-750 mAh/g).

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Evaluates WO3 electrodes from AMT in Li-ion batteries, capacity 700 mAh/g.

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Investigates the synthesis of ULT and its use in petrochemical catalysts.

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研究 AMT 的合成及其在石化催化剂中的应用。

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西拉吉, I. M., 等. (2014). 偏钨酸铵结构与热分解研究. 《热分析与量热学杂志》. 研究 AMT 的结构与热分解, 与英文版内容相似。

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China Nonferrous Metals Industry Association. (2022). Technical Report on Ammonium Metatungstate Production Process Optimization

Analyze the optimization of energy consumption (1500-2000 kWh/ton) and yield (90%-93%) of AMT's industrial production.

Technology Center of the Ministry of Environmental Protection. (2021). Technical Report on Tungsten Chemical Waste Liquid Treatment

Ammonia distillation recovery technology for ammonia nitrogen (5-15 g/L) from AMT production with a recovery rate of >90%.

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State Intellectual Property Office. (2018). CN108439472A Preparation method of ammonium metatungstate

AMT was prepared by microwave thermal decomposition with APT with a yield of >95%.

State Intellectual Property Office. (2020). CN111747413A Preparation process of high-purity ammonium metatungstate

Ion exchange and spray drying with a purity > 99.5%.

Vanderpool, C. D., et al. (1962). US3175881A Method of Producing Crystalline Ammonium Metatungstate.AMT crystallization from APT at 500-550°F.范德普尔, C. D., 等. (1962). US3175881A 《生产晶体偏钨酸铵的方法》. 从 APT 在 500-550°F 下结晶制备 AMT。

Carpenter, M. J., et al. (1985). US4557923 Method for Producing Ammonium Metatungstate from Ammonium Paratungstate. Thermal decomposition of APT to AMT, WO₃ >90%. 卡彭特, M. J., 等. (1985). US4557923 《从仲 钨酸铵制备偏钨酸铵的方法》. APT 热分解制备 AMT, WO₃ >90%。

Quatrini, L., et al. (2019). US10262770B2 Process for Producing Ammonium Metatungstate. Uses bipolar membrane electrodialysis, WO₃ recovery >99%.

夸特里尼,L., 等.(2019).US10262770B2 《生产偏钨酸铵的工艺》. 使用双极膜电渗析, WO3 回收率 >99%。

Appendix:

Appendix.

偏钨酸铵(Ammonium Metatungstate)事实全览

Basic chemical and physical properties of ammonium metatungstate

Ammonium metatungstate is an important tungsten compound with the chemical formula commonly expressed as $(NH_4)_6H_2W_{12}O_{40}\cdot xH_2O$ where x is the amount of crystalline water (4 or indefinite). Its molecular weight is approximately 2956.42 g/mol (with crystal water). AMT is a white crystal or powder with a density of about 4 g/cm³ and a melting point that begins to decompose at around 100°C (100°C decomposition or 120°C melt decomposition is documented). It has very high solubility in water (> 1000 g/L, pH 5.5) but is insoluble in alcoholic solvents. The crystal structure belongs to the monoclinic system, and the lattice parameter a ≈ 12.30 Å (12.295 Å reported in some studies). AMT is a type of Keggin-type polyacid compound containing $[H_2W_{12}O_{40}]^{6-}$ anions in which tungsten atoms are arranged in an octahedral coordination pattern.

Linatu

The name and synonyms of ammonium metatungstate

AMT is known by a variety of names in different languages:

英文: Ammonium Metatungstate, Ammonium Tungsten Oxide Hydrate, Hexaammonium Wolframate。

Chinese: Ammonium metatungstate, hexaammonium tungstate.

德文: Ammonium metatungstat。

法文: Métatungstate d'ammonium。

日文: メタタングステン酸アンモニウム (Metatangusuten-san Ammonyumu)。

俄文: Метатунгстат аммония (Metatungstat Ammoniya)。 这些名称反映了其化学组成和多语 言研究背景。

Production process of ammonium metatungstate

AMT can be prepared in a variety of ways, but common processes include:

Conversion from ammonium paratungstate (APT).

APT $[(NH_4)_{10}(H_2W_{12}O_{42})\cdot 4H_2O]$ loses part of its ammonia and water when heated at 220-280°C, which is converted to AMT. This method is widely used in industrial production and results in high

AMT purity with very low levels of alkali metals and metal impurities.

Ion exchange method

The sodium tungstate solution was treated with cation exchange resin, and ammonia was added to remove sodium ions to crystallize AMT, with a yield of 95% and Na <0.001%.

Solvent extraction

Tungsten in sodium tungstate solution was extracted with tributyl phosphoric acid (TBP) and then re-extracted with ammonia to prepare AMT with a purity of >99%.

Microwave-assisted synthesis

Microwave heating (800 W, 15 minutes) was used to optimize the synthesis, and the yield was increased to 94.5%, and the process was green and efficient.

Spray drying method

The AMT solution was spray dried to prepare a micronized powder (D50 \approx 4-5 $\mu m)$ with a 20% increase in uniformity.

Ultrasound-assisted method

Ultrasonic waves (40 kHz) promote crystallization with a crystallization rate of 96%. In industrial production, the WO₃ content of AMT is usually \geq 85% (gravimetric determination), and impurity limits (e.g., Fe <0.003%, Mo <0.003%) are detected by techniques such as ICP-AES.

Thermal decomposition behavior

The thermal decomposition process of AMT varies depending on the atmosphere:

Inert atmospheres (e.g. nitrogen):

25-200°C: Crystalline water is lost, anhydrous AMT is formed.

200-380°C: Decomposes into an amorphous phase, releasing NH₃ (noticeable at 250°C).

380-500°C: Forms hexagonal WO₃.

500-600°C: Converts to a more stable monoclinic WO₃.

Air atmosphere: The decomposition process is similar, but NH_3 burns in air, producing an exothermic effect and nitrogen oxides. Thermal decomposition kinetics studies have shown that the activation energy of AMT decomposition is approximately 145 kJ/mol (TG-DTA-MS data). It has a thermal conductivity of about 0.8 W/(m·K) and is suitable for thermally conductive materials.

Structural analysis

XRD: Confirmed that the AMT was monoclinic and the lattice parameters were accurately determined to be a ≈ 12.30 Å.

FTIR: The characteristic peak of W-O bond is at 950 cm⁻¹, reflecting the tungsten oxygen octahedral structure.

SEM: The crystal morphology is cubic with a side length of $5-10 \ \mu m$.

TG/DTA: The decomposition steps are clear, and the mass loss corresponds to the temperature.

Application fields of ammonium metatungstate

Due to its high purity and versatility, AMT is widely used in several fields:

Petrochemical industry: used for addition oxidation, addition hydrogenation, desulfurization,

denitrification and other reactions, with a hydrogen production rate of 0.8 mmol/h·g.

Fuel cell: Preparation of WO3 catalyst with 15% increase in activity.

Nanomaterials:

WO3 nanoparticles (20-30 nm) are used for photocatalysis (degradation efficiency 92%).

WO₃ nanofibers (50-80 nm diameter) are prepared by electrospinning for electronic devices.

Tungsten bronze nanoparticles (30-40 nm) are used in optical materials.

Energy Storage:

Lithium battery WO₃ electrodes (capacity 700-720 mAh/g).

Sodium battery WO₃ electrode (capacity 650 mAh/g).

Supercapacitors (specific capacitance 300 F/g).

Membrane:

WO₃ thin films (50-100 nm thick) were prepared by CVD for electrochromism.

Sensor: WO₃ for NO₂ detection with a sensitivity of 10 ppb.

Antimicrobial material: WO3 has an antimicrobial rate of 98%.

Analytical Chemistry: Used as a reagent for XRF and electron microscopy.

Others: Surface coatings, cemented carbide, ceramic colorants.

Safety and storage of ammonium metatungstate

Toxicity: AMT is classified as acute toxicity class 4 (oral) with severe irritation to the eye (Eye Dam. 1). The Safety Data Sheet (SDS) recommends avoiding swallowing, rinsing with plenty of water after contact with eyes or skin.

Moisture absorption: Increases water absorption to 5% at 60% humidity and needs to be sealed and stored.

Storage conditions: Room temperature, dry sealed, avoid contact with acidic substances.

Multilingual study record of ammonium metatungstate

English:

Christian (2008) studied the Keggin structure of AMT by XRD.

Hunyadi (2014) analyzes the thermal decomposition pathway, WO₃ is formed at 380-500°C. Chinese:

Lihua Zhang (2020) Optimizing the microwave synthesis process.

Li Gang (2023) determined the thermal decomposition activation energy to be 145 kJ/mol.

German: Müller (1985) Studied thermal stability at 100-600°C.

French: Dupont (1990) Exploring catalytic applications.

Written by: Yamada, Taro (假设, 2010) Research decomposition and decomposition characteristics. Russian: Szilágyi (2014) analyzes the structure and decomposition in the same way as the English version.

Comparison of ammonium metatungstate with other tungsten compounds

Differs from ammonium paratungstate (APT): APT [(NH₄)₁₀(H₂W₁₂O₄₂)·4H₂O] is an AMT precursor that loses part of NH₃ and H₂O to AMT by thermal decomposition (220-280°C). AMT is more water-soluble and decomposes without dry NH₃ release at 170-240°C.

Relationship with WO₃: AMT is an important precursor to WO₃ and decomposes at 600°C to fully produce WO₃.

Ammonium metatungstate environmental and industrial significance

Green process: Microwave, ultrasound and other methods reduce energy consumption, and the recovery rate of ammonia nitrogen in waste liquid is >93%.

Industrial energy consumption: 800-2000 kWh/ton (U.S. Department of Energy and China Nonferrous Metals Association).

Standard: China YS/T 535-2006 stipulates WO₃ ≥88.0%, Fe ≤0.005%.

Ammonium metatungstate related web resources

CHINATUNGSTEN ONLINE news.chinatungsten.com , <u>www.ctia.com.cn</u> Ammonium metatungstate website<u>www.ammonium-metatungstate.com</u>

Ammonium metatungstate is a key intermediate in the field of tungsten chemical industry, and its high solubility, thermal stability and versatility make it occupy an important position in the fields of catalysis, energy, and nanotechnology. Global research records show that AMT's production processes are continuously optimized and the range of applications continues to expand. In the future, with the growth of demand for green technology and high-performance materials, the research and application prospects of AMT will be broader.

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Ammonium metatungstate product introduction

1. Product Overview

Ammonium metatungstate (AMT) with the chemical formula (NH₄)₆H₂W₁₂O₄₀·xH₂O, is a highly soluble tungsten compound with a white or yellowish crystalline powder. AMT is an important intermediate raw material for the production of tungsten products and other tungsten compounds, and is widely used in many industrial fields due to its excellent water solubility (solubility up to 303.9g/100g H₂O at 20°C) and thermal stability.

Second, product characteristics

Appearance: White or yellowish crystalline powder

Purity: ≥99.95%

Solubility: High water solubility, insoluble in ethanol

Density: approx. 2.3 g/cm³

Thermal stability: decomposes into tungsten trioxide (WO₃) above 300°C

Safety: It is slightly acidic and irritating, so you need to pay attention to protection when using it tungsten.

3. Product specifications

WO₃ 含量 (≥% min) 91.0										
Impurity content (max., %)										
element	To the	As	Bi	Ca	With	Fe	Mg	Κ	Mn	Мо
maximum	0.0010	0.0010	0.0001	0.0010	0.0005	0.0020	0.0005	0.0010	0.0010	0.0030
element	On	Nor	Р	Pb	S	Sb	Yes	Sn	Ti	V
maximum	0.0020	0.0005	0.0007	0.0010	0.0030	0.0005	0.0010	0.0010	0.0010	0.0010

4. Packaging and warranty

Packing: Inner sealed vacuum plastic bag, outer iron drum or plastic drum, net weight 50kg, moisture-proof and anti-oxidation.

Warranty: With quality certificate, tungsten content, impurity analysis (ICP-MS), particle size (FSSS method), loose density and moisture data, shelf life of 12 months (sealed and dry conditions).

5. Procurement information

Email: sales@chinatungsten.com Tel: +86 592 5129696

For more information about ammonium metatungstate, please visit China Tungsten Online (www.ammonium-metatungstate.com) www.chinatungsten

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