Application note



Enhancing quality control with automated impurity detection in $LiFePO_4$ cathode materials

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The importance of impurity control in battery materials manufacturing

Lithium iron phosphate ($LiFePO_4$ or LFP) cathode materials have become a critical part of lithium-ion battery (LIB) manufacturing due to their superior thermal stability, long cycle life, enhanced safety, and cost-effectiveness.[1] Their widespread adoption in electric vehicles and energy storage systems has driven rapid expansion of LFP manufacturing worldwide, accounting for an estimated 60 to 65% of global cell production.[2] As production volumes continue to increase, it is becoming increasingly critical to maintain consistent raw material quality and clean manufacturing processes to help ensure battery performance, reliability, and manufacturing yield.

When mass-producing LFP cathode materials, detecting and controlling particulate impurities is a critical quality challenge. These impurities, which can compromise product quality and process consistency, can originate from raw materials, processing equipment wear, or the manufacturing environment. Depending on their composition and size, impurities may increase manufacturing variability, adversely impact electrochemical performance, and, in severe cases, create defects that may lead to internal short circuits and battery safety risks.[3] Consequently, impurity analysis has become an important component of routine quality control in battery materials.

The growing importance of impurity control is reflected in the development of industry standards. For example, in China, GB/T 41704-2022 formalizes a standardized method for testing the magnetic impurity of LIB cathode materials. Additionally, a metallic-impurity standard currently under development for LFP and other cathode materials is expected to expand the range of impurities analyzed to include Fe, Cr, Ni, Cu, and Zn using a combination of ICP-OES, optical microscopy (OM), and scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS). This demonstrates an increasing emphasis on identifying not only the quantity of impurities but also their morphology and chemical composition.

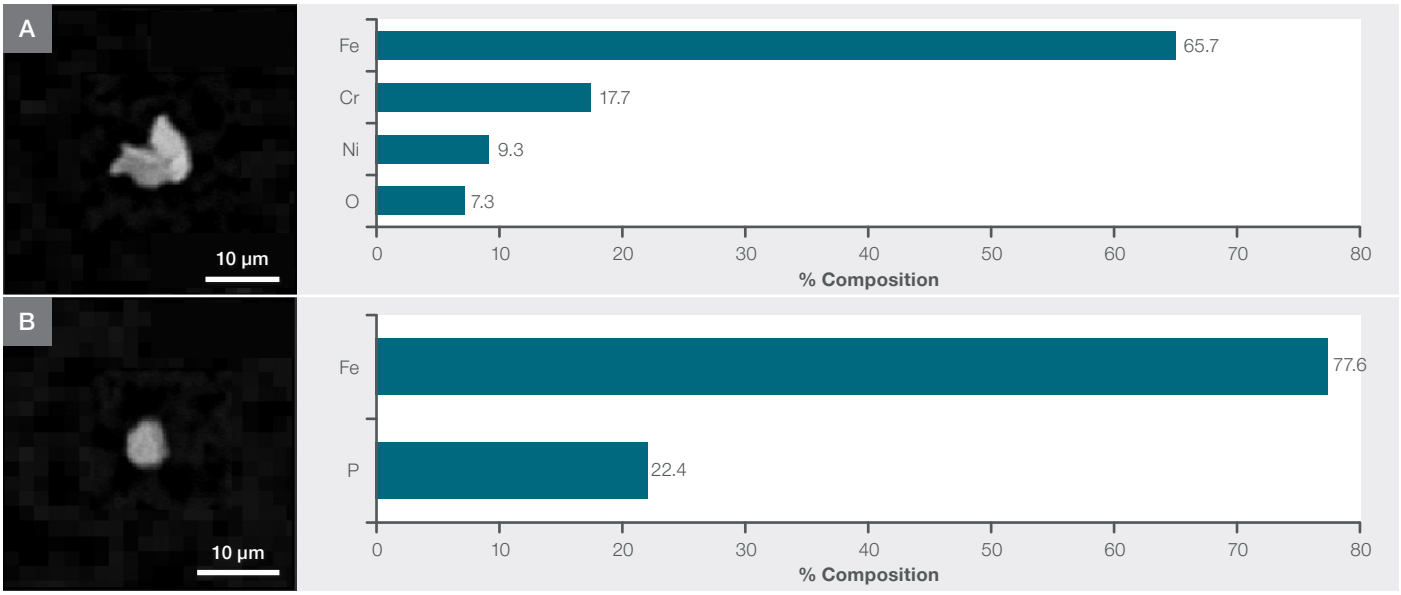


Figure 1. Analysis of a Fe-Cr-Ni-rich particle (A) and a Fe-P-rich particle (B).

For LFP materials, interpreting iron-containing particles requires particular attention because iron is both an intrinsic element of the cathode itself and a key element in the impurities. For example, a Fe-Cr-Ni-rich particle may suggest stainless-steel-like equipment wear, while a Fe-P-rich particle may be related to an LFP process secondary phase (Figure 1). Although both particles contain iron, they originate from different sources and require different root-cause investigations and corrective actions. An effective quality control method must therefore not only detect particles but also accurately and consistently classify them according to their elemental composition and size.

This application note explores an automated SEM-EDS workflow leveraging the Thermo Scientific™ Phenom™ ParticleX™ Battery Desktop SEM for particle detection and compositional classification in LFP cathode materials. By combining automated particle detection with elemental analysis, this workflow enables rapid, standardized identification of impurities, helping manufacturers improve the efficiency of quality control, support root-cause analysis, and maintain consistent production quality.

Existing methods and the information gap in current QC workflows

Battery manufacturers employ a range of analytical techniques to monitor and control contamination in cathode materials. ICP-based methods, for instance, can be leveraged for sensitive quantification of trace elemental impurities, while X-ray fluorescence (XRF) can support fast elemental screening. Optical microscopy (OM), meanwhile, is widely used to detect, count, and size particles. However, it usually provides only broad particle grouping (e.g., metallic versus non-metallic particles) without revealing detailed compositional information.

Although these methods are valuable for bulk elemental analysis and initial screening, they largely cannot be used to fully answer particle-level questions, such as:

- What is the composition of an individual particle?
- Does the particle originate from stainless-steel wear, a synthesis process, copper/zinc contamination, or the LFP matrix?
- How does the particle impact product quality, and is it high risk?

SEM-EDS can answer such particle-specific questions by combining high-resolution imaging with elemental analysis. Individual particles can be characterized based on their size, morphology, and chemical composition, helping researchers classify and investigate the root cause of battery impurities. However, conventional manual SEM-EDS is labor-intensive, operator-dependent, and difficult to standardize for routine batch-level QC in battery manufacturing environments.

The Phenom ParticleX Battery Desktop SEM addresses these challenges by automating SEM-EDS particle analysis. Rather than replacing ICP, XRF, and OM, it complements these techniques with automated, reliable, and high-throughput analysis. The Phenom ParticleX Battery Desktop SEM can analyze approximately 20,000 to 30,000 particles per hour—revealing their morphology, elemental composition, and classification—and provide standardized reporting at the individual particle level. Compared to manual analysis, this comprehensive information supports faster impurity identification, more consistent quality control, and more efficient root-cause analysis for producing LFP cathode materials.

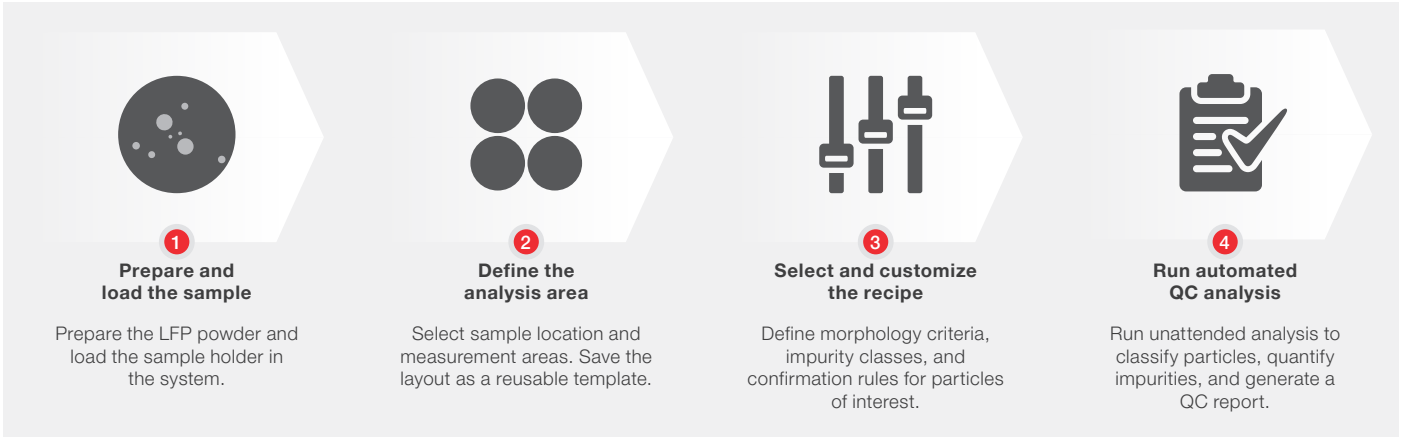


Figure 2. Automated Phenom ParticleX Battery Desktop SEM workflow for particle analysis, classification, and QC reporting.

A: Chemistry table (average chemical composition reported in %)

Classification	Particles	%	O	Al	Si	P	Ti	Cr	Mn	Fe	Ni	Cu	Zn	Zr	Mo
Fe-Cr-Ni-rich	265	21.5	13.5		0.1		0.2	14.8		61.5	8.0				
Fe-P-rich (low O)	383	31.1	11.8	0.1		15.1				69,5	0.9				
Zr-rich	12	1.0	38.0		3.3									55.1	
W-rich	3	0.2	23.5							6.1					9.1
Fe-rich	554	44.9	27.4		0.3					67.6					3.0
Zn-rich	3	0.2	8.4										64.9		26.7
Cu-rich	13	1.1	20.1								1.2	68.9			2.8

How Perception Software and the Phenom ParticleX Battery Desktop SEM work together to automate SEM-EDS particle analysis

The Phenom ParticleX Battery Desktop SEM workflow is designed specifically for particulate impurity analysis in battery materials. It reduces manual work and supports repeatable analysis through a recipe-based SEM-EDS method. A typical workflow (Figure 2) includes sample preparation, recipe setup, automated backscattered electron (BSE) imaging, EDS compositional analysis, rule-based classification, and automatic report generation.

An LFP sample was used to demonstrate this impurity analysis process. First, particles were concentrated and collected on a standardized 47 mm–diameter filter using a series of dispersion, filtration, separation, and matrix removal steps. Following sample preparation, a scan area and minimum particle size were defined in the recipe, along with BSE imaging and EDS acquisition settings, classification rules, and the desired reporting format. The Phenom ParticleX Battery Desktop SEM then automatically scanned the selected area using BSE imaging, detecting particles and measuring their size and morphology. EDS spectra were subsequently acquired for the detected particles to identify their elemental composition.

B: Particle limits

Rule class	Particle count	Minimum	Maximum
Cu-rich	13	0	10
Zn-rich	3	0	10
Fe-Cr-Ni-rich	265	0	300
Fe-P-rich (low O)	383	0	100
Fe-rich	554	0	100
W-rich	3	0	50
Zr-rich	12	0	500

C: Histogram chart generated based on Dmax

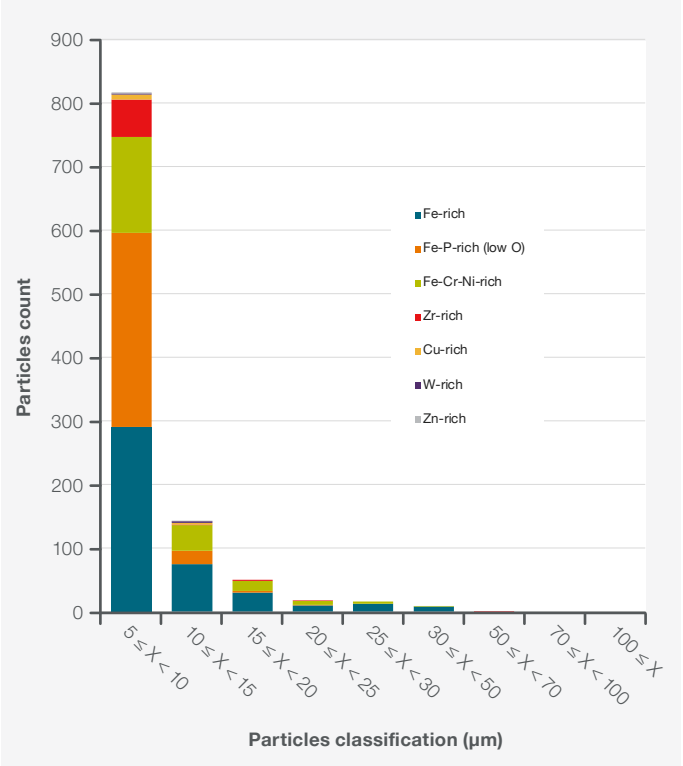


Figure 3. QC overview of particulate impurities: (A) chemical classification and average composition, (B) particle counts by impurity class compared with predefined acceptance limits, and (C) particle size distribution by chemical classification.

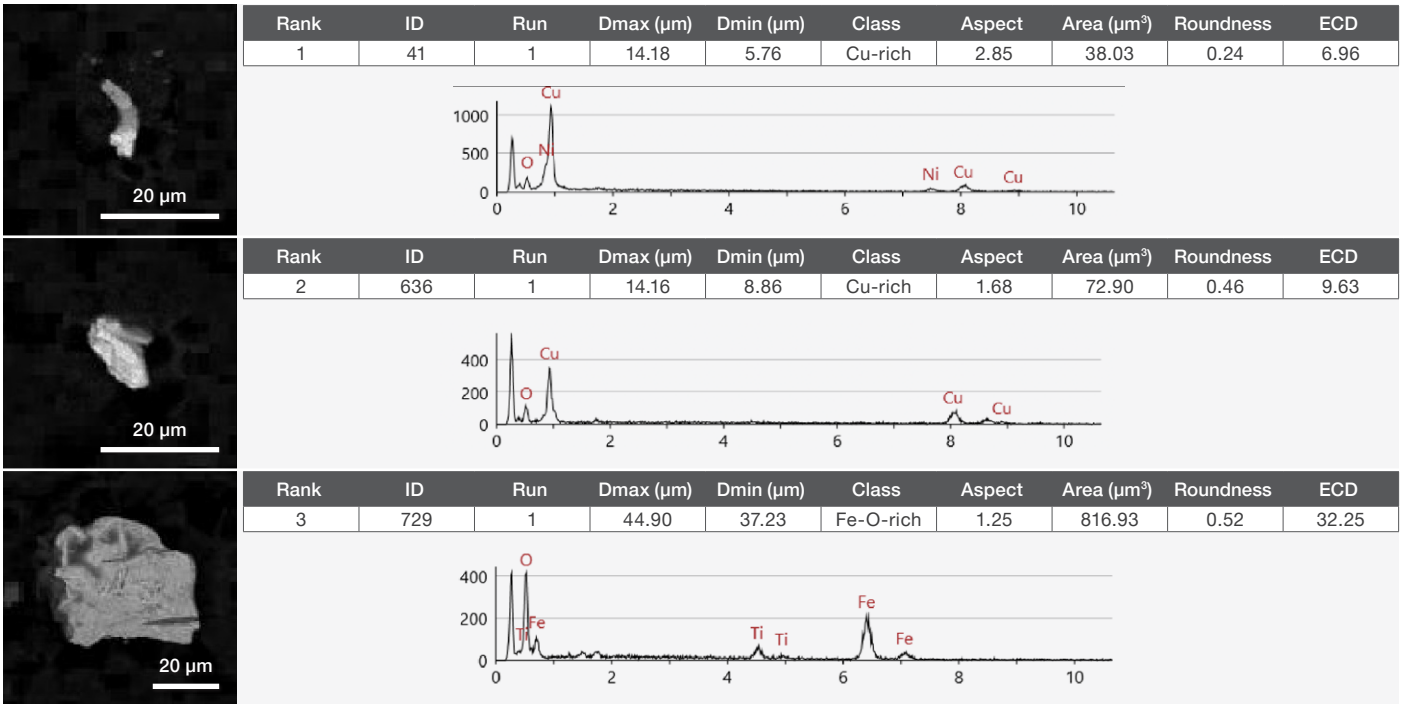


Figure 4. Example of particle-level verification in a report.

To systematically identify and classify potential impurities, particles were automatically categorized using predefined, editable rule templates based primarily on elemental composition. Here, particles were categorized as either LFP-matrix-like, Fe-P-rich, Fe-Cr-Ni-rich, Cu-rich, or unknown/review-required.

Following this analysis, the Thermo Scientific™ Perception Software automatically compiled results into standardized QC-ready reports using editable and reusable reporting templates. The example in Figure 3 combines a QC overview with particle-level verification, which helps you move from rapid batch assessment to detailed impurity confirmation and root-cause investigation.

Quality control overview

Within the report, the chemistry table (Figure 3A) summarizes impurity classes, particle counts, relative abundances, and the average elemental composition. In this example, iron-containing classes account for more than 80% of the classified particles, indicating that iron-related contamination is the main concern for this sample. The particle limits table (Figure 3B) compares measured counts with user-configurable acceptance limits based on internal QC requirements. This sample is marked as “FAIL” because multiple impurity classes exceed their specified maximum values. The Dmax histogram (Figure 3C) shows that the largest particle populations occur in the 5 to 10 µm and 10 to 15 µm ranges, indicating that the detected contamination is dominated by relatively fine particles. Together, these results can help battery manufacturers prioritize inspection of iron-bearing process-contact and material-handling components to evaluate whether corrective actions reduce impurity counts and particle sizes.

Particle-level verification

Each characterized particle is linked to an information table containing its identification, classification, dimensions, and key morphology measurements along with the corresponding SEM image and EDS spectrum (Figure 4). This data shows particle-level evidence that verifies the rule-based classification and helps relate recurring elemental and morphological signatures to potential raw-material, equipment, or handling sources.

Recipe-based analysis for supplier-to-cell quality alignment

In battery manufacturing, analytical throughput has an enormous impact because each material production batch may need quality verification. At the same time, the quality control method must also deliver accurate, repeatable, and standardized results that can be consistently aligned among material suppliers, cell manufacturers, QC laboratories, and production sites.

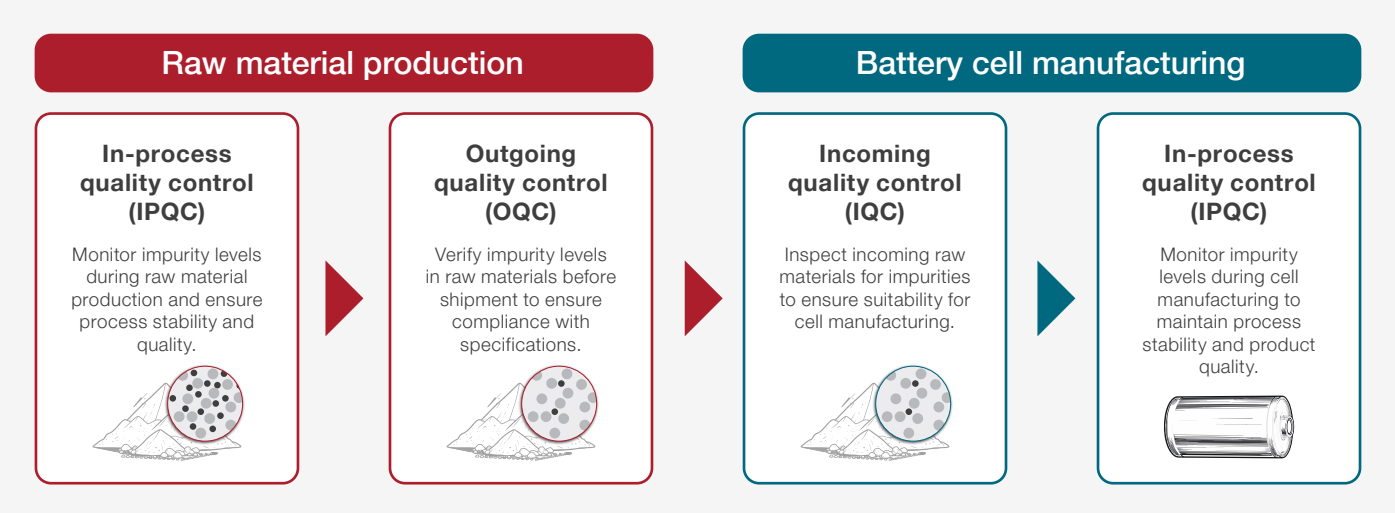


Figure 5: Role of impurity analysis across the battery manufacturing value chain.

One of the key strengths of the Phenom ParticleX Battery Desktop SEM is its recipe-based workflow, which standardizes particle analysis and reporting. A recipe can define a range of critical parameters, including the analysis area, minimum particle size, imaging and EDS acquisition conditions, classification rules, high-risk particle criteria, and the report template and data export format. Once established, the same validated recipe can be reused across multiple instruments, operators, and laboratories, minimizing operator variability and helping to ensure consistent analytical results. From incoming material qualification and in-process control to outgoing quality control, this standardized workflow supports consistent quality assessment and alignment across the battery value chain (Figure 5). For example, a cathode material supplier can use a recipe for outgoing QC and a cell manufacturer can use the same recipe for incoming material inspection. Likewise, a third-party laboratory can use the same classification rules for abnormal batch review or independent verification. This creates a shared analytical language across the supply chain.

This approach also follows the quality-chain logic used in technical cleanliness standards such as ISO 16232 and VDA 19.1, which emphasize using documented, repeatable, transferable, and comparable analytical methods between upstream and downstream users for effective quality assurance.

For impurity analysis in LFP cathode materials, this consistency is especially important. A dedicated recipe helps consistently classify particles across operators, laboratories, suppliers, and cell manufacturers to support a more reliable quality control process.

Practical considerations for routine, automated particle analysis

Routine quality control in battery manufacturing requires more than analytical capability. To facilitate high-volume production, the particle analysis workflow must also be reliable, stable, and cost-effective, thereby supporting continuous operation in both QC and factory labs.

The Phenom ParticleX Battery Desktop SEM is designed to support routine, automated particle analysis by minimizing manual intervention, standardizing recipe-based workflows, and generating automated, QC-ready reports. Key features that support efficient day-to-day operation include:

- **High-throughput automated analysis:** Up to 30,000 particles per hour can be processed (depending on sample particle density), supporting unattended, high-volume daily testing
- **Long-life CeB_6 SEM source:** With an operating lifetime of up to 3,000 hours, the CeB_6 source supports reliable 24/7 QC operation, reduces maintenance frequency, and consequently lowers the analytical cost per sample
- **Optimized hardware design:** Long component lifetimes support stable and reliable analysis
- **Automated, recipe-based workflows:** Standardized analysis recipes improve reproducibility across operators, instruments, and laboratories while reducing user-dependent variability
- **Software for consistent data acquisition:** Software improvements, such as an AI-based particle segmentation option, help improve data consistency during automated runs
- **Compact SEM platform:** The small footprint of Phenom Desktop SEMs allows them to fit in a variety of spaces, including QC and factory labs, as well as near-line manufacturing environments
- **Advanced EDS analysis:** Optimized EDS algorithms support complex particle identification in battery materials

These features of the Phenom ParticleX Battery Desktop SEM make automated SEM-EDS particle analysis more practical, supporting routine, repeated batch analysis, long unattended automated runs, supplier comparisons, and impurity monitoring throughout the production of $LiFePO_4$ cathode materials.

Conclusions

Effective impurity control is essential for maintaining the quality, consistency, and reliability of LFP cathode materials. However, no single analytical technique can provide a complete assessment, as contamination can originate from multiple sources and can exist in different forms. Established methods can contribute valuable information for quality control but ultimately cannot obtain complete, per-particle insights. The Phenom ParticleX Battery Desktop SEM complements these techniques by offering automated SEM-EDS-based particle characterization, which can reveal particle-level information including size, morphology, elemental composition, and classification, while also supporting standardized reporting.

As demonstrated for a battery QC workflow, the Phenom ParticleX Battery Desktop SEM can help you move from labor-intensive, slow, manual characterization to automated, high-throughput, recipe-based particle analysis. A standardized workflow supports batch inspection, consistent quality alignment across suppliers and manufacturers, and efficient root-cause analysis, ultimately supporting continuous process improvement in the production of LFP cathode materials. By offering automated and standardized particle-level SEM-EDS characterization, the Phenom ParticleX Battery Desktop SEM supports production of safer, higher-quality lithium-ion batteries.

References

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