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Beyond Peak Matching: X-Ray Diffraction, Phase Identification, and Mineralogical Uncertainty in Electric Arc Furnace Dust — A Critical Review

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Abstract

Electric arc furnace dust (EAFD) is a heterogeneous metallurgical residue in which bulk chemistry alone cannot resolve the chemical form, crystal structure, substitution state, or accessibility of Zn, Fe, and minor elements. X-ray diffraction (XRD) is therefore central to EAFD characterization, but the apparent simplicity of phase search–match can conceal substantial uncertainty arising from peak overlap, solid solutions, nanocrystallinity, microabsorption, preferred orientation, poorly crystalline material, and incomplete structural models. This critical review evaluates the reliability of qualitative and quantitative XRD for EAFD, with emphasis on zincite, zinc ferrite/franklinite-type spinels, magnetite–maghemite discrimination, XRD-amorphous/unmodeled material, and phase evolution during thermal and hydrometallurgical treatment. Particular attention is given to Rietveld-based quantitative phase analysis, the meaning of fit quality and uncertainty, and the role of SEM–EDS, EPMA/WDS, Mössbauer spectroscopy, Raman spectroscopy, X-ray absorption methods, and automated mineralogy in validating phase assignments. The review argues that a diffraction peak compatible with $ZnFe_2O_4$ does not, by itself, demonstrate stoichiometric zinc ferrite, and that process predictions are only as defensible as the mineralogical assignments they are based on. We propose a practical evidence hierarchy and multimethod workflow to connect phase identification with reagent demand, zinc recovery, ferrite refractoriness, thermal transformation, and residue behavior. The resulting framework supports more transparent reporting, uncertainty-aware mineralogical mass balances, and stronger translation from XRD patterns to metallurgical decisions.

Keywords: electric arc furnace dust; X-ray diffraction; Rietveld refinement; zinc ferrite; spinel solid solutions; process mineralogy

Highlights

- XRD phase matching alone cannot establish the chemistry of substituted EAFD spinels.
- Rietveld results require explicit treatment of amorphous content, microabsorption, orientation, and uncertainty.
- Multimethod validation is essential for magnetite–maghemite and Zn–Fe–Mn–Cr spinel discrimination.

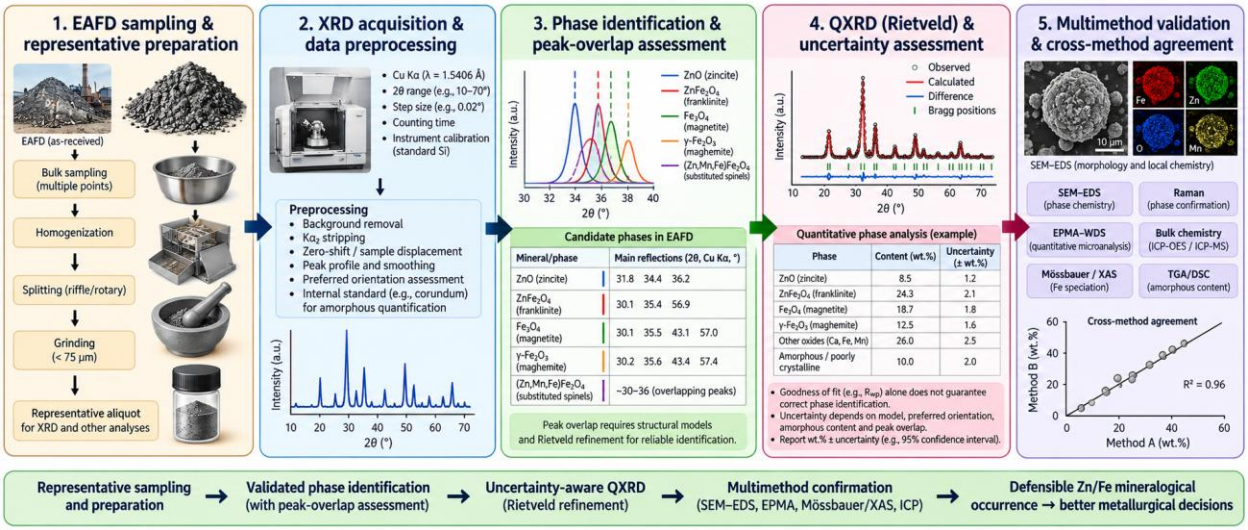
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- Reliable phase identification materially changes leaching, reduction, roasting, and residue interpretations.

Graphical Abstract



1. Introduction

Electric arc furnace dust forms through volatilization, condensation, mechanical entrainment, and secondary reactions in steelmaking off-gas systems. Consequently, particles collected in a single baghouse can combine primary oxide fragments, high-temperature condensates, transformed droplets, salts, carbonaceous matter, and phases inherited from scrap, fluxes, refractories, and alloying additions. This heterogeneity explains why total Zn or Fe does not directly reveal how those elements will behave during washing, leaching, reduction, roasting, or smelting (Abadi & Tang, 2024; Al-Harahsheh et al., 2019; Branca et al., 2020; Chen et al., 2011; Cherrat et al., 2025; De Buzin et al., 2017; He et al., 2026; Huang et al., 2024).

XRD is the most widely used structural tool for moving from elemental composition to mineralogical interpretation, yet EAFD is an unusually demanding matrix. ZnO may be relatively distinctive, whereas cubic Zn–Fe spinels, magnetite, maghemite, Mn-bearing spinels, Cr-bearing spinels, and compositionally substituted ferrites can produce overlapping or nearly coincident reflections. In addition, fine condensates and reaction products may be poorly

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crystalline, and high-Z constituents intensify microabsorption effects. A pattern can therefore be consistent with a phase without uniquely proving its stoichiometry (Machitouen et al., 2026; Pickles, 2010; Tang et al., 2022; Wu et al., 2014).

This review asks: how reliably can XRD identify and quantify mineral phases in EAFD, and what analytical strategies are required to distinguish structurally similar and compositionally substituted phases? The manuscript treats phase identification as an evidence problem rather than as a database-matching exercise. Figure 1 frames EAFD complexity, Figure 2 locates uncertainty along the interpretation chain, Figure 3 illustrates the spinel-overlap problem, Figure 4 summarizes major QXRD uncertainty contributors, Figure 5 presents the proposed multimethod validation workflow, and Figure 6 connects phase identity to metallurgical behavior. Table 1 defines the evidence hierarchy, Table 2 summarizes phase-specific XRD confidence, Table 3 focuses on spinel discrimination, Table 4 lists dominant sources of analytical error, Table 5 maps complementary methods to unresolved questions, and Table 6 translates validated mineralogy into process implications (Aranda et al., 2012; Black et al., 2011; Cline et al., 2015; David, 2010; Dinnebier & Leineweber, 2018; Dinnebier & Billinge, 2008).

Figure 1 should be read as a reminder that the XRD problem begins before diffraction: the analytical target is a multiscale material in which bulk chemistry, crystallinity, substitution, and particle-scale association are coupled.

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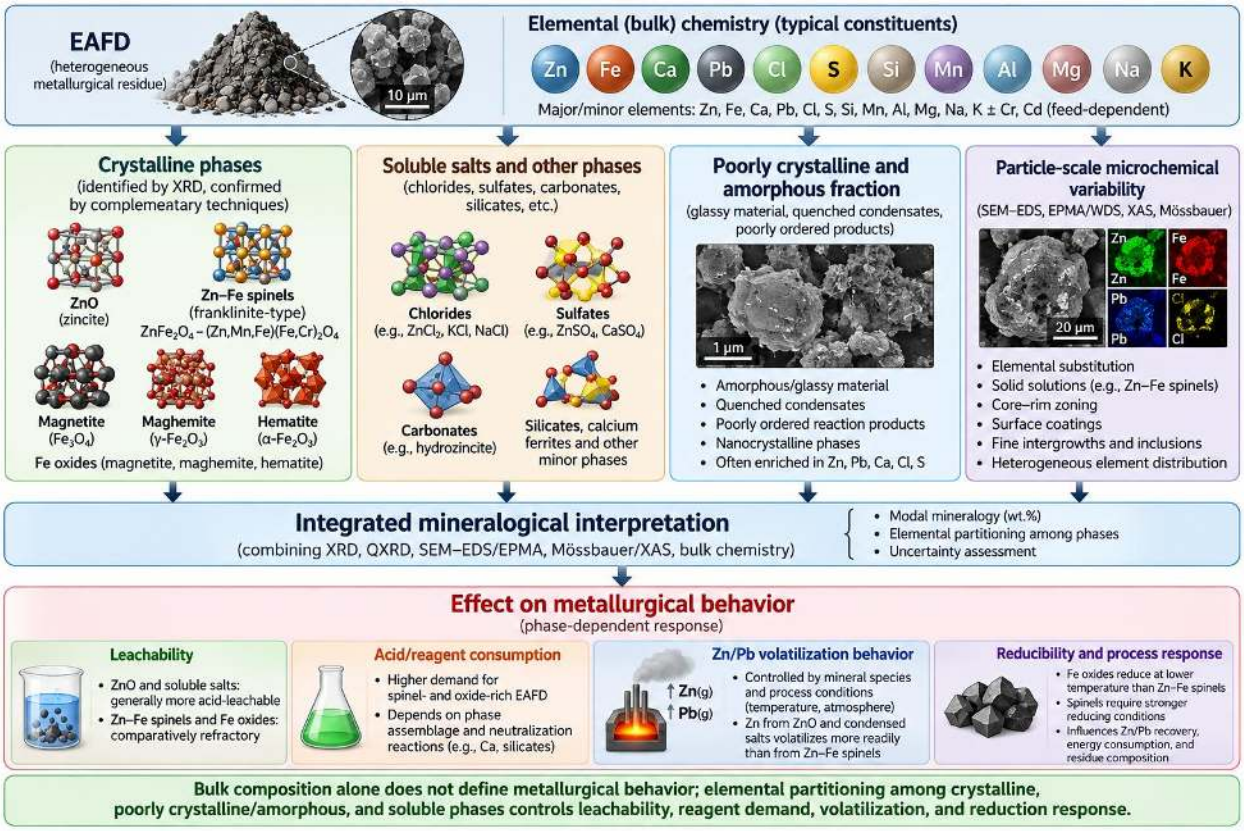


Figure 1. Multiscale complexity of EAFD as a diffraction and process-mineralogy problem.

Note. Adapted from an original conceptual synthesis based on the EAFD characterization and recycling literature reviewed in this article.

The layers in Figure 1 imply that a defensible mineralogical statement must distinguish what diffraction measures directly from what is inferred from composition, morphology, or process response.

2. Scope and Critical Review Methodology

2.1. Review scope

The review covers EAFD from carbon and stainless steelmaking, including fresh, aged, and processed materials; qualitative XRD; quantitative phase analysis; Rietveld refinement; internal- and external-standard approaches; in situ or high-temperature diffraction; and complementary compositional, spectroscopic, and microscopic methods. The emphasis is not on

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cataloging every reported phase, but on evaluating the strength of evidence supporting phase assignments and the metallurgical consequences of uncertainty (Abadi & Tang, 2024; Al-Harahsheh et al., 2019; Aranda et al., 2012; Binnemans & Jones, 2020; Brandner & Antrekowitsch, 2021; De Buzin et al., 2017).

2.2. Search logic and evidence screening

This article is a critical, evidence-weighted narrative review rather than a formal systematic review or meta-analysis. Google Scholar was used as the primary literature-discovery platform, with publisher webpages, DOI records, and bibliographic databases used to verify metadata and final publication status. The core search window was 2010–2026, and the literature corpus and bibliographic metadata were last updated on 2 October 2026. Representative Boolean combinations included: ("electric arc furnace dust" OR EAFD) AND (XRD OR "X-ray diffraction"); (EAFD OR "electric arc furnace dust") AND (Rietveld OR "quantitative phase analysis"); (EAFD OR "electric arc furnace dust") AND (zincite OR "zinc ferrite" OR franklinite OR spinel); (EAFD OR "electric arc furnace dust") AND (magnetite OR maghemite OR hematite) AND (XRD OR Mössbauer OR XANES); (EAFD OR "electric arc furnace dust") AND (amorphous OR nanocrystalline OR microabsorption OR "preferred orientation"); and (EAFD OR "electric arc furnace dust") AND (SEM-EDS OR EPMA OR WDS OR Raman OR XPS OR XANES OR EXAFS OR "automated mineralogy").

Eligible sources comprised peer-reviewed journal articles, authoritative books and book chapters, standards or reference-material publications, and selected technical reports that contributed directly to EAFD mineralogy, diffraction methodology, phase validation, phase-transformation mechanisms, or metallurgical interpretation. Preprints, records lacking traceable publication metadata, purely descriptive papers without relevant mineralogical evidence, duplicate records, and sources whose analytical principles were not transferable to EAFD were excluded. English-language sources were prioritized; non-English studies were eligible only when sufficient English bibliographic or methodological information was available for critical

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appraisal. Methodological studies on other complex multiphase materials were retained only when the analytical principle was directly transferable to EAFD.

Screening was performed by title, abstract, and then full-text relevance where available. Backward reference checking of seminal diffraction and process-mineralogy papers and forward citation tracing were used to identify methodological sources that keyword searches could miss. The final corpus comprised 138 references after duplicate removal and relevance screening. Evidence was weighted according to analytical specificity, independence and orthogonality of corroborating methods, quantitative uncertainty, and relevance to the metallurgical decision; database search–match alone was treated as screening evidence rather than definitive phase confirmation. Because no prospective PRISMA log was maintained, no retrospective PRISMA flow diagram was reconstructed.

2.3. Evidence hierarchy

Table 1 presents an author-developed evidence hierarchy used as a decision-support synthesis in this review. It is not a validated analytical standard, and it does not imply that every study must progress through all levels or use every technique. Analytical sufficiency is question-dependent: confidence should increase when independent, orthogonal evidence addresses the specific ambiguity relevant to the metallurgical decision, whereas additional but redundant measurements do not necessarily increase evidentiary strength.

Table 1. Evidence hierarchy for mineral-phase assignment in EAFD.

Level	Minimum evidence	Typical strength	Principal limitation
I	XRD search–match only	Screening	Susceptible to overlap and database misassignment
II	XRD + bulk chemistry/XRF	Contextual	Bulk composition does not prove phase chemistry
III	XRD + SEM–EDS	Moderate	EDS interaction volume may mix fine phases
IV	QXRD/Rietveld + standards/uncertainty	Strong for abundance	Dependent on model completeness and bias control
V	XRD + EPMA/WDS or phase-	Strong for	Higher analytical effort

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	sensitive spectroscopy	chemistry/speciation	
VI	Integrated XRD + QXRD + microanalysis + spectroscopy + mass balance	Integrated / highest evidentiary convergence	Resource intensive; value depends on orthogonality and decision relevance

Note. This author-developed hierarchy is an unvalidated decision-support synthesis. Evidence strength refers to decision-specific reliability of phase identity and abundance, not to a universal ranking of analytical techniques; progression is risk-driven rather than mandatory.

In practice, Level I may be adequate for rapid screening of a dominant, well-resolved phase, but it is weak evidence for compositionally variable spinels. Higher-convergence evidence is warranted when a phase assignment controls mass-balance closure, kinetic interpretation, reagent selection, reduction or roasting conditions, residue stability, or a claim of selective recovery. A carefully designed XRD–Mössbauer experiment can therefore be more decision-sufficient for a specific Fe-phase question than a broader but redundant multimethod campaign (Aranda et al., 2012; Bish, 2013; Black et al., 2011; Christidis et al., 2020; Cline et al., 2015; Coelho, 2018; David, 2010; De la Torre et al., 2010).

3. Mineralogical Complexity of EAFD as an XRD Problem

3.1. Major, minor, and poorly crystalline constituents

Commonly reported crystalline constituents include zincite (ZnO), Zn-bearing spinels often labeled $ZnFe_2O_4$ or franklinite, magnetite, hematite, silica, Ca-bearing phases, chlorides, sulfates, and minor Pb-, Mn-, and Cr-bearing compounds. Their relative abundance depends on scrap composition, furnace practice, gas cooling, dust recirculation, and downstream conditioning. The same nominal EAFD therefore cannot be assumed to have the same mineralogical response across plants or campaigns (Lin et al., 2017; Gargul et al., 2021; Matsukevich & Kulinich, 2022; Pereira & Fonseca, 2025; Rieger et al., 2021; Sajal & Ahmad, 2025; Thottempudi et al., 2022; Trifunović et al., 2022; Wang et al., 2021).

Poorly crystalline and nanocrystalline condensates are analytically important because their weak and broadened diffraction features can be absorbed into background, misfit, or neighboring peaks. Material not represented by the crystalline model can comprise at least five analytically distinct categories: known crystalline phases, poorly crystalline or nanocrystalline

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phases, structurally disordered material, unidentified or inadequately modeled crystalline contributions, and genuinely XRD-amorphous material. These categories should not be collapsed automatically into an 'amorphous' fraction. When the experiment cannot resolve them, the more defensible term is 'XRD-amorphous/unmodeled fraction.' Failure to account for this fraction can make crystalline phase percentages appear internally precise while excluding material that may contain significant Zn, Fe, Cl, or other elements (Christidis et al., 2020; De la Torre et al., 2010; Itakura et al., 2025; Jansen et al., 2011; Kern & Madsen, 2012; Madsen & Scarlett, 2011; Mittemeijer & Welzel, 2008).

3.2. Solid solutions and compositional substitution

The term 'zinc ferrite' is often used operationally for a cubic spinel-like phase, but EAFD spinels may incorporate Mn, Mg, Al, Cr, and other cations. Such substitution changes lattice parameters, site occupancy, redox state, and potentially leachability or reducibility. Accordingly, structural identity and chemical identity should remain formally separated as structure type → compositionally constrained phase → chemically validated phase. Where Mn occupancy and phase chemistry are not independently established, 'franklinite-type' or 'Zn–Fe–Mn-bearing spinel' is more defensible than unqualified 'franklinite.' The structural similarity that makes spinels stable at high temperature also makes them difficult to discriminate by routine powder diffraction alone (Machitouen et al., 2026; Pickles, 2010; Tang et al., 2022; Wu et al., 2014).

Figure 2 shows why phase identification should be treated as an analytical chain. Later, more sophisticated refinement does not correct errors introduced during sampling or grinding.

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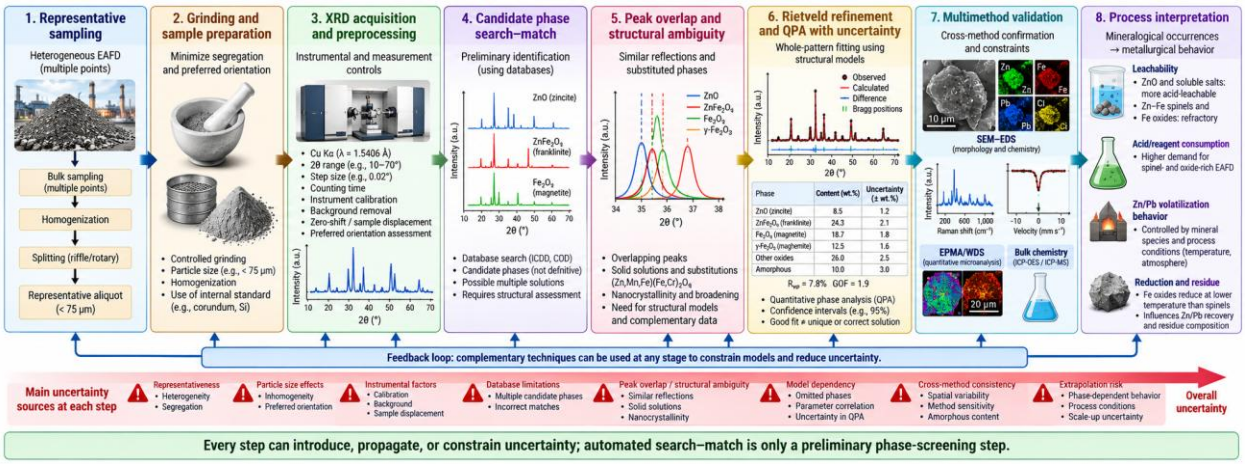


Figure 2. Analytical chain from sampling to metallurgical interpretation.

Note. Adapted from an original synthesis of powder-diffraction quality-control principles and EAFD process-mineralogy practice reviewed in this article.

A critical implication of Figure 2 is that refinement software cannot compensate for a non-representative sample, severe microabsorption, missing structural models, or phase chemistry that has not been independently constrained.

4. Fundamentals of XRD Applied to EAFD

4.1. Diffraction position, intensity, and breadth

For crystalline phases, Bragg diffraction follows $n\lambda = 2d \sin\theta$. Peak position therefore reflects lattice spacing and can reveal structural shifts from substitution, whereas intensity depends on structure factors, multiplicity, preferred orientation, absorption, and instrumental geometry. Peak breadth contains contributions from crystallite size, microstrain, defects, and instrumental response. For EAFD, these quantities are not merely textbook descriptors: they determine whether closely related spinels can be separated and whether weak minor phases remain detectable (Balzar, 2011; Chipera et al., 2013; Cornell et al., 2013; Dvořák et al., 2017; Fan et al., 2026; He, 2018; Hill et al., 2010; Holder et al., 2019).

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4.2. Detection limits and database dependence

A phase can be absent from a reported XRD result because it is truly absent, below the practical detection threshold, poorly crystalline, or masked by stronger phases. Search–match quality also depends on the reference database and on whether the candidate phase model reflects the composition actually present. High similarity scores are therefore not proof of chemical identity (Gates-Rector et al., 2019; Chateigner et al., 2012; Lee et al., 2020; Lee et al., 2021; Prasianakis, 2024; Schuetzke et al., 2021; Szymanski et al., 2023).

4.3. Why the fundamentals matter specifically for EAFD

EAFD combines strong absorbers, very fine particles, broad size distributions, and multiple oxide families. These conditions make specimen displacement, fluorescence, microabsorption, preferred orientation, and statistical noise especially consequential. The relevant question is not whether XRD can detect crystalline phases—it can—but whether the experimental and modeling conditions support the specificity implied by the reported mineral names (Chateigner et al., 2012; Lee et al., 2020; Lee et al., 2021; Schuetzke et al., 2021; Szymanski et al., 2023).

5. Identification of Zinc-Bearing Phases

5.1. Zincite

ZnO is often among the more readily recognized Zn-bearing phases because its wurtzite structure provides a distinctive set of reflections. Even so, quantitative abundance may be biased by particle statistics, absorption contrast, preferred orientation, and incomplete accounting of amorphous material. A strong ZnO signal should therefore be interpreted as evidence of a major crystalline Zn reservoir, not as a direct measure of total leachable Zn (Gamutan et al., 2024; Kalpakli et al., 2021; Li & Wang, 2010; Toporkova & Prodanova, 2020; Zoraga et al., 2022).

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5.2. Zn-bearing spinels and non-spinel Zn phases

Zn associated with cubic spinels is substantially more difficult to interpret. Reports of $ZnFe_2O_4$, franklinite-type phases, or related Zn–Fe–Mn spinels must be considered against the possibility of continuous substitution and overlapping reflections. Conversely, soluble or volatile Zn-bearing chlorides and sulfates may be minor in diffraction intensity but disproportionately important in washing, chloride management, sulfation roasting, or downstream purification (Chen et al., 2011; Huang et al., 2024; Pereira & Santos, 2025b; Tang et al., 2022; Toporkova & Prodanova, 2020; Wu et al., 2014).

Table 2 summarizes the main Zn- and Fe-bearing phase families and the confidence that conventional XRD alone can usually provide.

Table 2. Phase-specific interpretation of conventional XRD in EAFD.

Phase/family	**Typical XRD confidence**	**Main ambiguity**	**Recommended confirmation**
ZnO (zincite)	High when abundant	Overlap/minor broadening	SEM–EDS; QXRD
ZnFe2O4-like spinel	Moderate	Substitution; overlap with other spinels	EPMA/WDS; Mössbauer/XAS; refined lattice parameter
Franklinite-type spinel	Moderate	Zn–Fe–Mn compositional range	EPMA/WDS; XAS
Magnetite	Moderate	Maghemite/spinel overlap	Mössbauer; Raman; magnetic methods
Hematite	High–moderate	Low abundance/broadening	Mössbauer or Raman if critical
Chlorides/sulfates	Moderate–low when minor	Hydration, deliquescence, weak abundance	IC/chemical extraction; SEM–EDS
Amorphous/disordered fraction	Low by XRD alone	Hidden mass and metals	Internal/external standard; chemical mass balance

Note. Confidence assumes adequate sampling and data quality; downgrade it when peaks are weak, broad, or heavily overlapped.

The table reinforces an important distinction: a mineralogical label should communicate the specificity of the evidence. Where XRD supports only a spinel-structure assignment,

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reporting 'Zn-bearing spinel' may be more defensible than reporting a pure stoichiometric $ZnFe_2O_4$ phase.

6. The Spinel Identification Problem

6.1. $ZnFe_2O_4$ versus magnetite, maghemite, and franklinite-type phases

Cubic spinels share closely related reflection families. In EAFD, the analytical problem is intensified because cation substitution can shift peaks by amounts comparable to instrumental broadening, while nanoscale domains broaden reflections and multiphase mixtures create additional overlap. Magnetite and maghemite can also be difficult to separate when present with Zn-bearing spinels, especially at low abundance (Machitouen et al., 2026; Pickles, 2010; Tang et al., 2022; Wu et al., 2014).

Figure 3 uses synthetic patterns to illustrate the interpretive problem: two structurally similar spinels can produce a sequence of peaks that appears nearly coincident at routine resolution.

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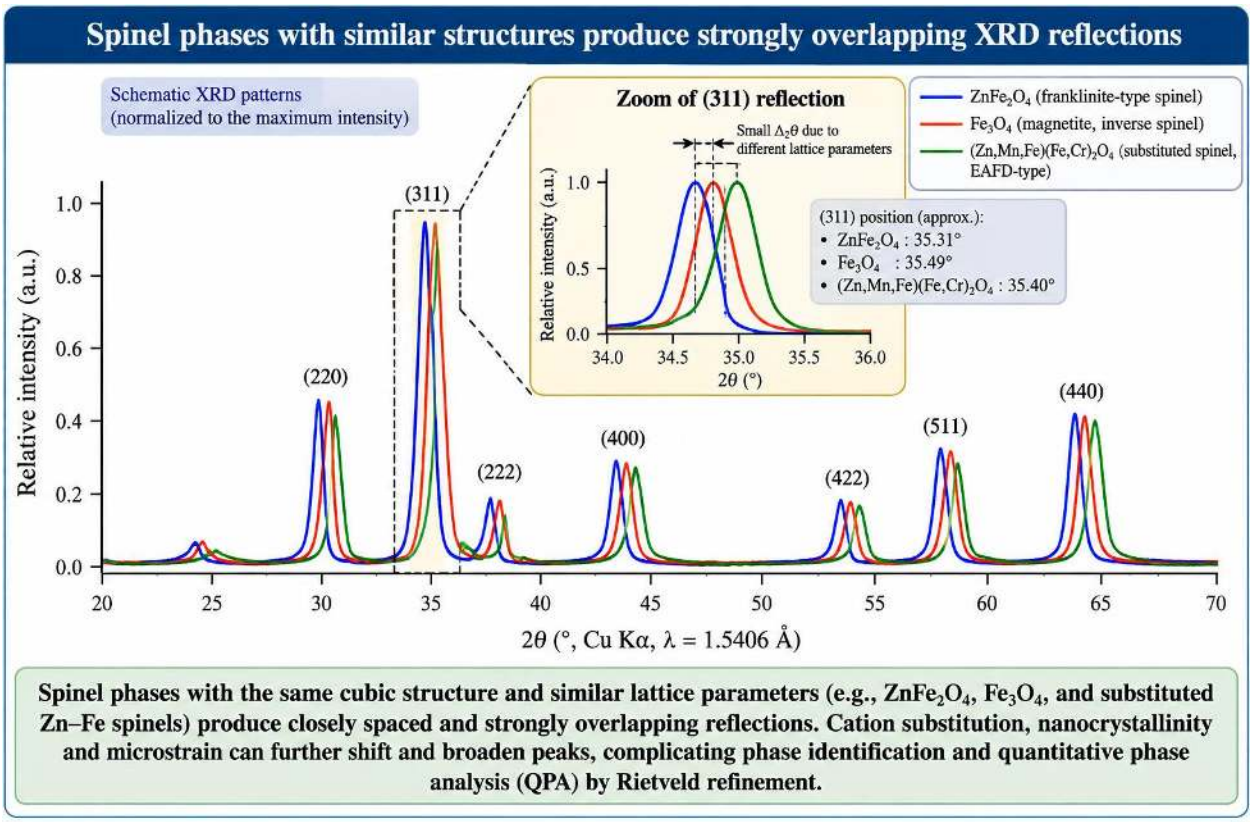


Figure 3. Schematic overlap of diffraction reflections from structurally similar spinel phases.

Note. Adapted as an original schematic from spinel crystallography and EAFD phase-identification issues discussed in the cited literature; intensities and positions are illustrative, not reference patterns.

6.2. Lattice parameter is informative but not sufficient

Refined lattice parameters can provide evidence of substitution, but lattice constant alone generally cannot infer a unique composition because multiple substitution vectors can produce similar shifts. Therefore, constrain phase chemistry through microanalysis or element-specific spectroscopy whenever the distinction affects process interpretation (Machitouen et al., 2026; Pickles, 2010; Meena & Balraju, 2025; Tang et al., 2022; Wu et al., 2014).

6.3. Reporting discipline for spinels

When the evidence is limited to overlapping cubic reflections, the report should distinguish structural family from chemical formula. Terms such as 'spinel-type phase' or 'Zn-

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bearing spinel consistent with a $ZnFe_2O_4$-rich composition' are more transparent than an unqualified stoichiometric assignment. Table 3 provides a practical decision framework.

Table 3. Decision framework for reporting spinel-like phases in EAFD.

Observed evidence	Defensible wording	What is still unresolved
Search–match only; strong overlap	Spinel-type phase	Cation chemistry and exact phase identity
XRD + Zn/Fe-rich SEM–EDS association	Zn–Fe-bearing spinel	Stoichiometry; Mn/Cr/Mg/Al substitution
Rietveld + lattice parameter + EPMA/WDS	Compositionally constrained Zn–Fe spinel	Site occupancy/redox may remain uncertain
XRD + microanalysis + Mössbauer/XAS	Validated Zn–Fe spinel assignment	Residual uncertainty should be reported quantitatively

Note. The wording should not imply greater chemical specificity than the analytical evidence supports.

This discipline is particularly important in critical reviews because apparently minor naming differences can propagate into mechanistic claims about refractoriness, reduction temperature, or selective leaching (Machitouen et al., 2026; Orozco-Henao et al., 2022; Tang et al., 2022; Wu et al., 2014).

7. Iron-Bearing Phases and Their Identification

7.1. Magnetite, hematite, maghemite, wüstite, and metallic Fe

Fe-bearing phases record both furnace redox history and subsequent transformation. Hematite is often comparatively straightforward when crystalline and abundant, whereas magnetite–maghemite discrimination can be problematic in complex mixtures. Wüstite and metallic Fe may occur in reduced or partially processed materials, and their presence can strongly affect acid consumption, galvanic reactions, and reduction/smelting behavior (Cornell et al., 2013; Hiraga & Gomes, 2021; Orozco-Henao et al., 2022; Qiu et al., 2026; Wu et al., 2014; Zhang et al., 2014).

7.2. The special value of Mössbauer and Fe K-edge methods

Because XRD primarily constrains crystal structure, Fe-specific techniques can resolve oxidation state and site occupancy when diffraction peaks are ambiguous. Mössbauer

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spectroscopy is especially useful for distinguishing Fe-bearing magnetic and paramagnetic environments, whereas Fe K-edge XANES can constrain oxidation state and coordination. Their value is highest when the analytical question is not simply 'is an Fe oxide present?' but 'which Fe-bearing structure controls the observed process response?' (Fleet, 2013; Gütlich & Bill, 2011; Orozco-Henao et al., 2022; Meena & Balraju, 2025; Wilke et al., 2011)

8. Quantitative Phase Analysis and Rietveld Refinement

8.1. From qualitative identification to phase fractions

Rietveld refinement fits the full diffraction pattern using structural models, scale factors, peak-shape functions, background terms, lattice parameters, and other variables. In EAFD, this is potentially transformative because it converts a descriptive pattern into a mineralogical mass-balance component. However, a numerical phase fraction is only as reliable as the sample, models, absorption corrections, amorphous treatment, and constraints used in the refinement (Aranda et al., 2012; Bish, 2013; Black et al., 2011; Christidis et al., 2020; Cline et al., 2015; Coelho, 2018; David, 2010; De la Torre et al., 2010; Dinnebier & Leineweber, 2018; Dinnebier & Billinge, 2008; Eberl, 2010; Gates-Rector et al., 2019).

8.2. Amorphous content and unknown structures

Internal- and external-standard strategies can estimate the XRD-amorphous/unmodeled fraction, while PONKCS-type approaches can incorporate phases for which complete crystal structures are unavailable. Internal-standard QXRD requires control of spike mass, standard crystallinity, mixing completeness, particle-size compatibility, and microabsorption between the standard and EAFD phases; otherwise the standard itself can introduce bias. External-standard approaches avoid physically adding a standard but depend strongly on instrument stability, calibrated scale relationships, identical or demonstrably comparable acquisition conditions, and appropriate reference measurements. PONKCS-type results can improve quantitative closure for incompletely modeled phases, but they do not by themselves establish chemically definitive phase identity. For EAFD, these distinctions matter because normalizing only modeled

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crystalline phases to 100% can hide a substantial XRD-amorphous/unmodeled fraction and create apparently precise but systematically biased phase percentages (Christidis et al., 2020; De la Torre et al., 2010; Itakura et al., 2025; Jansen et al., 2011; Kern & Madsen, 2012; Madsen & Scarlett, 2011; Westphal et al., 2012).

8.3. Fit indices are not uncertainty statements

Rwp, goodness-of-fit, and visual residuals are useful diagnostics, but none alone demonstrates correct phase identity or an unbiased phase fraction. A refinement can fit well with compensating model errors, incorrect minor phases, or an inadequately modeled background. Software-derived standard uncertainties represent only part of the measurement uncertainty. Where the result is decision-critical, reporting should include independent specimen preparations, repeated scans where practical, alternate plausible phase models, sensitivity to background and peak-shape choices, treatment of the XRD-amorphous/unmodeled fraction, and the effect of model choice on the reported phase fractions. Between-preparation variability is particularly important because sample heterogeneity can exceed least-squares uncertainty (Gualtieri et al., 2019; León-Reina et al., 2014; Madsen & Scarlett, 2011; Rowles, 2021; Toby, 2006).

Figure 4 summarizes the uncertainty contributors that EAFD QXRD should consider explicitly.

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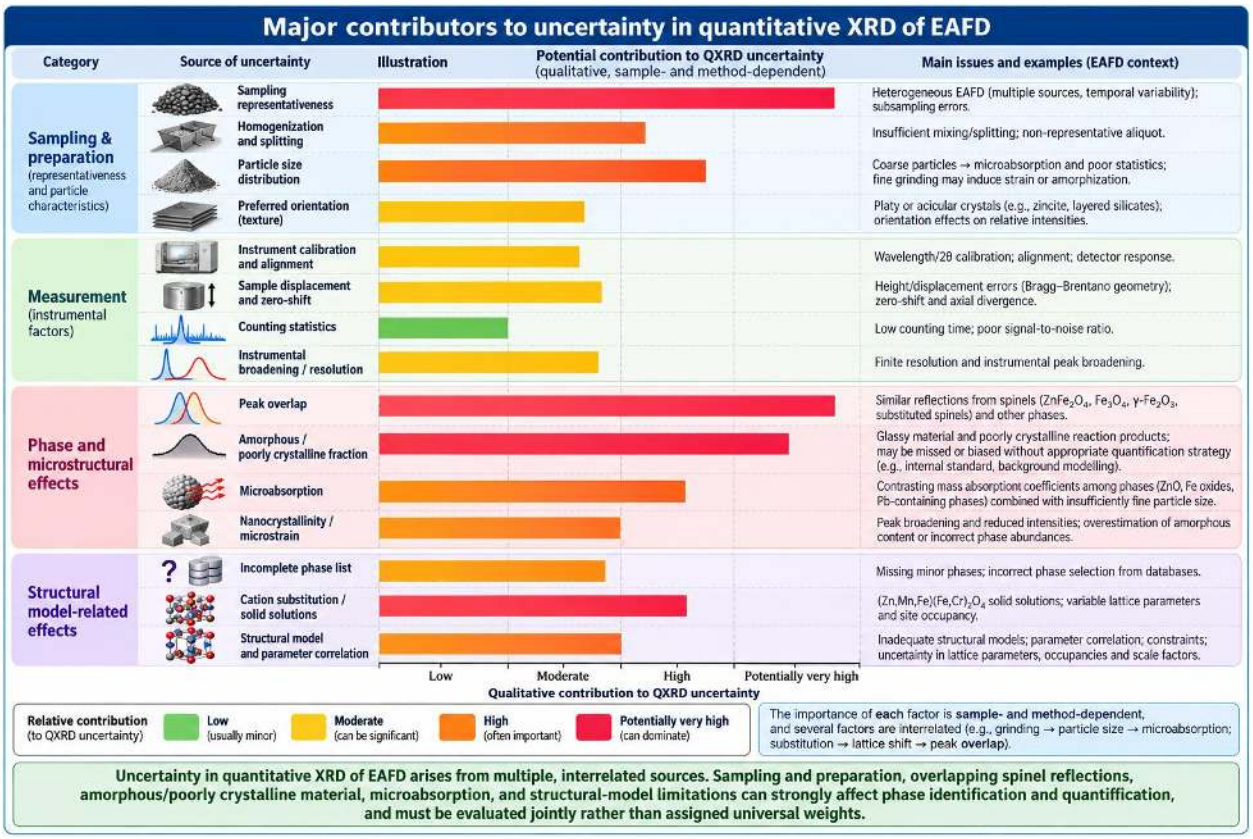


Figure 4. Conceptual criticality of major uncertainty contributors in quantitative XRD of EAFD.

Note. Adapted from an original synthesis of QXRD/Rietveld error sources discussed across standards, round-robin studies, and complex-material applications in the reviewed literature.

The relative scale in Figure 4 is conceptual rather than metrological; it prevents the false inference that software-generated standard errors capture all sources of uncertainty.

9. Sources of Error in EAFD XRD Analysis

9.1. Sampling, grinding, preferred orientation, and microabsorption

EAFD is often finer than conventional mineral powders, but fine particle size does not automatically guarantee representative powder statistics. Agglomeration, segregated dense particles, soluble salts, and metallic fragments can undermine aliquot representativity. Sample state must therefore be treated as part of the uncertainty chain. Reports should identify sampling location, storage atmosphere, storage duration and time between collection and XRD, drying

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conditions, washing history, humidity exposure, and grinding or milling protocol. This is essential because hygroscopic chlorides, sulfates, carbonates, hydrated phases, and other metastable constituents can change during storage or preparation. Grinding can improve homogeneity but may alter soluble or hydrated phases and can broaden peaks. Preferred orientation is phase-dependent, while microabsorption becomes important when phases differ strongly in absorption coefficient and particle size. Where relevant, studies should report approximate particle sizes, qualitative or quantitative absorption contrast, specimen homogenization, and whether a Brindley-type correction or sensitivity analysis was applied (Hill et al., 2010; Kleeberg & Monecke, 2010; Lopresti et al., 2022; Shen et al., 2025).

9.2. Fluorescence, peak overlap, broadening, and low abundance

Fe-rich materials measured with Cu radiation can suffer Fe fluorescence that degrades the signal-to-background ratio unless instrumental mitigation is used, for example an appropriate monochromator, energy-discriminating detector, alternative radiation, or other validated configuration. Reproducible EAFD XRD reporting should state the radiation source and wavelength, monochromator or detector configuration, diffraction geometry, scan range, step size, counting time, specimen spinning where applicable, calibration/reference standard, and instrumental-broadening model. Peak broadening from small crystallites or strain further reduces specificity, and weak minor phases can disappear beneath major-peak tails. The practical detection threshold is therefore experiment- and matrix-specific rather than a universal concentration percentage (He, 2018; Hill et al., 2010; Mittemeijer & Welzel, 2008; Prescher et al., 2015; Scardi & Leoni, 2010).

9.3. Database misassignment and uncritical automation

Automated search–match and machine-learning tools can accelerate pattern analysis, but they do not eliminate the need for chemical plausibility, reference-pattern quality, and expert review. Training data may not represent substituted industrial spinels, and an algorithm optimized for phase labels can still return a chemically misleading name. Automation is most valuable when embedded in a workflow that exposes confidence, candidate alternatives, and

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independent validation (Chateigner et al., 2012; Lee et al., 2020; Lee et al., 2021; Schuetzke et al., 2021; Szymanski et al., 2023).

Table 4 converts the principal error mechanisms into practical controls.

Table 4. Major sources of error in EAFD XRD and recommended controls.

Error source	Likely effect	Diagnostic sign	Recommended control
Non-representative aliquot	Biased phase fractions	Poor replicate agreement	Composite sampling; replicate preparations
Insufficient homogenization	Particle-statistics bias	Variable intensities	Controlled milling/mixing
Preferred orientation	Distorted intensities	Systematic reflection mismatch	Back-loading/side-loading; orientation model
Microabsorption	Biased scale factors	Composition-dependent residuals	Fine uniform powder; correction/sensitivity test
Fluorescence/background	Higher detection limit	Low signal-to-background	Appropriate radiation/optics; longer counting
Peak overlap	False or unstable phase split	Strong parameter correlation	Constrain chemistry; complementary methods
Nanocrystallinity/strain	Broad peaks	Poor narrow-peak models	Size–strain model; TEM if critical
Database/model error	False phase assignment	Good match but implausible chemistry	Check reference data; alternative models
Unmodelled amorphous phase	Inflated crystalline percentages	Chemical mass-balance closure failure	Internal/external standard

Note. Controls should be selected according to the decision risk associated with the reported mineralogical result.

A strong analytical report should state not only the phases that were found, but also the plausible phases that could not be uniquely separated and the controls used to reduce those ambiguities.

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10. Complementary Techniques for Phase Validation

10.1. SEM–EDS, EPMA/WDS, and automated mineralogy

SEM–EDS can test whether Zn, Fe, Mn, Cr, Ca, Pb, Cl, and S occur in the same particles or in distinct populations, while EPMA/WDS provides substantially stronger quantitative constraints on individual crystalline domains. For fine intergrowths, the EDS interaction volume can mix adjacent phases, so SEM–EDS association should not be treated as precise phase stoichiometry. Automated mineralogy can add particle-scale modal and association information, but elemental similarity, segmentation, and reference-library classification do not independently prove crystal structure; in particular, automated SEM-based classification should not be treated as direct confirmation of magnetite versus maghemite or closely related spinels without independent structural constraints. These methods address a core limitation of XRD: diffraction identifies long-range structures, whereas process interpretation often requires phase chemistry and liberation/association (Fandrich et al., 2007; Goldstein et al., 2018; Gottlieb et al., 2010; Reed, 2011).

10.2. Mössbauer, Raman, XPS, and X-ray absorption spectroscopy

Mössbauer spectroscopy is particularly powerful for Fe-bearing phases and oxidation states. Raman spectroscopy can distinguish selected oxide polymorphs and provide local structural information, while XPS is explicitly surface-sensitive and may disproportionately represent altered particle surfaces; it should not be interpreted as a bulk phase-fraction measurement. XANES/EXAFS provides element-specific local coordination and oxidation-state evidence even when long-range crystallinity is weak. Mössbauer and XAS are orthogonal and powerful, but their spectral deconvolution and model selection can themselves be non-unique. Complementary evidence therefore reduces uncertainty rather than necessarily eliminating it, and spatial-scale or sampling mismatches among methods must also be considered. No single technique is universally superior; the unresolved hypothesis should determine the complementary method (Fleet, 2013; Goldstein et al., 2018; Gottlieb et al., 2010; Gütlich et al.,

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2011; Nasdala et al., 2012; Orozco-Henao et al., 2022; Potts & West, 2011; Reed, 2011; Meena et al., 2025; Wilke et al., 2011).

Figure 5 presents a decision-oriented multimethod workflow rather than a fixed analytical recipe.

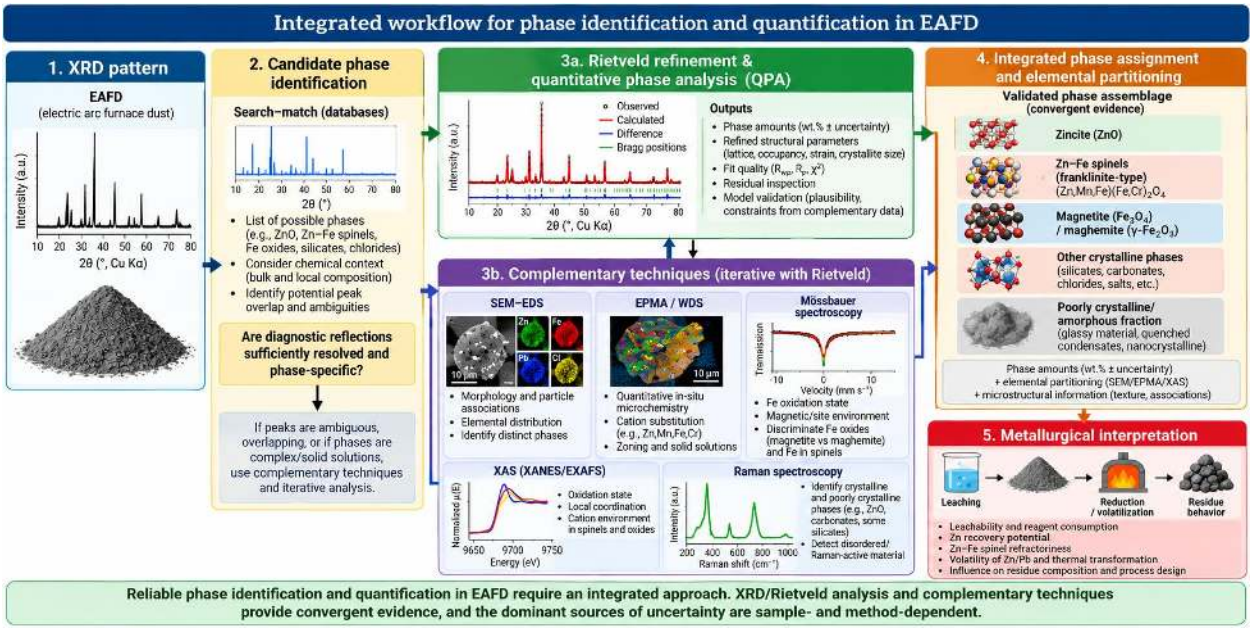


Figure 5. Multimethod workflow for validating uncertain EAFD phase assignments.

Note. Adapted from an original synthesis of diffraction, microscopy, microanalysis, and spectroscopy roles described in the reviewed literature.

The workflow prioritizes escalation: well-resolved major phases may require only QXRD and routine microchemical checks, whereas ambiguous spinels or Fe oxidation-state questions justify element-specific validation.

Table 5 maps common unresolved questions to the complementary methods most likely to reduce uncertainty.

Table 5. Complementary methods for resolving XRD ambiguities in EAFD.

Unresolved question	**Most useful complementary method(s)**	**Information added**
Is the spinel Zn–Fe, Zn–Fe–Mn,	EPMA/WDS; SEM–EDS	Phase chemistry and substitution

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or Cr-bearing?	mapping; XAS	
Magnetite or maghemite?	Mössbauer; Raman; magnetic characterization	Fe valence/site and polymorph discrimination
Where are Cl and S located?	SEM–EDS/WDS; ion analysis; XPS/XAS where appropriate	Particle association and chemical state
Is weak XRD signal due to amorphous material?	Standard-based QXRD; TEM/SAED; spectroscopy	Disordered fraction and local structure
Does the identified phase host the target metal?	EPMA/WDS; automated mineralogy	Element-to-phase allocation
Does feed mineralogy explain residue behavior?	Matched feed/residue QXRD + chemistry	Mineralogical mass balance and transformation

Note. Method selection should be hypothesis-driven; redundant measurements are less valuable than orthogonal evidence.

11. XRD During Thermal and Chemical Transformations

11.1. Reduction, calcination, and roasting

Static feed characterization is insufficient when EAFD treatment deliberately changes phase assemblage. Carbothermic, hydrogen-assisted, microwave, lime-modified, and other thermal routes transform Zn- and Fe-bearing phases through reduction, volatilization, oxidation, and solid-state reaction. XRD of staged products can identify disappearance of ZnO, evolution or decomposition of spinels, changes in Fe oxides, and the appearance of metallic or secondary phases (Ahmad et al., 2022; Anzulevich et al., 2021; Brandner & Antrekowitsch, 2021; Chang et al., 2022; Chen et al., 2023; Fan et al., 2026; Grudinsky et al., 2022; Hamann et al., 2024; Ju et al., 2025; Li et al., 2023; Ma et al., 2022; Omran & Fabritius, 2019; Qiu et al., 2026; Wu et al., 2023).

11.2. In situ and high-temperature XRD

In situ or high-temperature XRD can distinguish transient transformations that staged ex situ measurements may miss. A defensible nonambient experiment should report reaction atmosphere and gas flow, heating rate, temperature calibration at the specimen position, dwell time, time resolution, sample geometry, and any limitations imposed by windows or nonambient cells. Gas–solid reactions can be strongly path-dependent, and ex situ quenching may preserve metastable products, reverse transformations during cooling, or miss short-lived intermediates. In

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situ measurements can reduce these quench artifacts, but they introduce their own geometric, absorption, background, and temperature-gradient constraints; therefore nonambient data should be interpreted within the actual measurement configuration rather than as an artifact-free reference state.

11.3. Sulfation, chlorination, and salt-mediated transformations

Sulfation roasting aims to redirect Zn into water- or acid-soluble sulfate while limiting Fe dissolution, whereas chlorination can volatilize Zn/Pb chlorides or alter phase accessibility. Because selective chemistry depends on which phase reacts, diffraction should be coupled to elemental balances and product/residue chemistry. Apparent Zn removal without phase tracking cannot distinguish direct volatilization from intermediate formation and subsequent decomposition (Chen et al., 2023; Hamann et al., 2024; Ju et al., 2025; Wu et al., 2023).

11.4. Leaching and residue mineralogy

Matched feed and residue characterization is essential for hydrometallurgical interpretation. ZnO dissolution, persistence of Zn-rich spinels, secondary precipitation, and Fe-bearing residue evolution can all be tested by comparing phase fractions before and after leaching. Without this comparison, a high extraction can be mistakenly attributed to a specific phase, or residual Zn can be assigned to ferrite without confirming the residue mineralogy (Bakkar, 2014; Binnemans & Jones, 2020; Fan et al., 2026; Gamutan et al., 2024; Halli et al., 2018; Halli et al., 2017; Kalpakli et al., 2021; Kinnunen et al., 2025; Kukurugya & Vindt, 2015; Kul et al., 2015; Lanzerstorfer et al., 2020; Li & Wang, 2010; Miranda & Iglesias-González, 2026; Montenegro et al., 2016).

The key analytical sequence is therefore feed mineralogy → phase transformation or selective dissolution → residue mineralogy → elemental mass-balance closure. This sequence converts XRD from descriptive characterization into a mechanistic process tool (Xiong et al., 2024; Xue et al., 2025; Yang et al., 2024; Zhang et al., 2014).

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12. From Phase Identification to Metallurgical Interpretation and Knowledge Gaps

12.1. Mineralogy as a predictor of process response

ZnO-rich EAFD generally offers greater direct leaching accessibility than spinel-rich material, whereas Zn incorporated into refractory Zn–Fe spinels often requires stronger chemical conditions or prior thermal transformation. Ca-bearing phases can raise acid demand, chloride/sulfate phases affect washing and solution purification, and Fe oxide/spinel assemblages influence redox response and residue stability. These relationships are probabilistic rather than deterministic: nominal phase identity does not fix process response, because substitution, defect density, grain size, coatings, porosity, liberation, and particle association also influence leachability and reducibility. The level of phase-identification confidence required should therefore be decision-dependent. A broad 'spinel present' assignment may be adequate for preliminary screening, but it is insufficient when the claim concerns ferrite-controlled Zn recovery, selective leaching mechanism, reduction temperature, residue stability, or metal deportment (Mousavi & Mousavi, 2024; Oustadakis et al., 2010; Palimąka et al., 2018; Pereira & Fonseca, 2026; Pereira & Santos, 2025a; Pereira & Santos, 2025b; Prasetyo et al., 2020; Teimouri et al., 2022; Terrones-Saeta et al., 2021; Toporkova & Prodanova, 2020; Trifunović et al., 2024; Yang et al., 2025; Zoraga et al., 2020; Zoraga et al., 2022).

Figure 6 summarizes the conceptual trade-off between process accessibility and interpretation uncertainty for representative phase families. The plotted axes are ordinal, non-validated conceptual scales only; they must not be read as calibrated 0–10 scores or as a validated process-accessibility metric.

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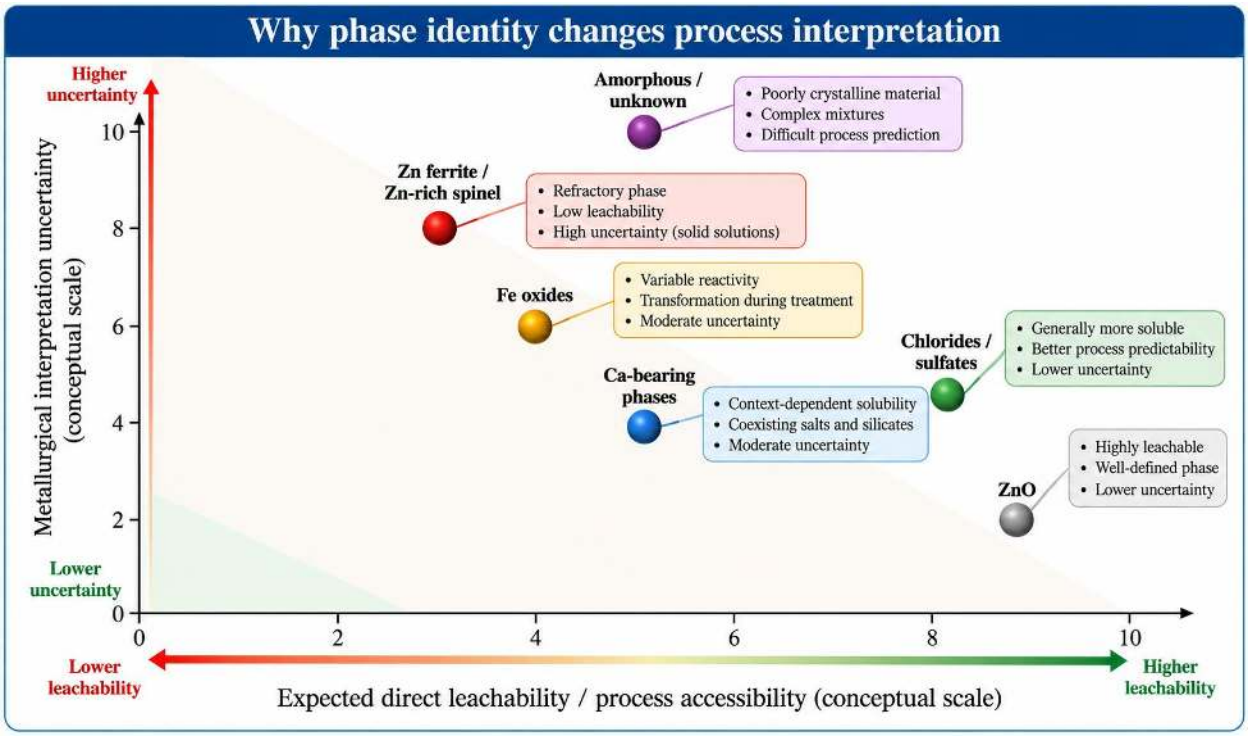


Figure 6. Conceptual relationship between phase identity, direct process accessibility, and mineralogical uncertainty using ordinal, non-validated conceptual axes.

Note. Adapted from an original synthesis of phase-specific leaching, reduction, roasting, and validation evidence reviewed in this article. Axis positions are qualitative (lower/intermediate/higher) and are not calibrated process scores.

The figure highlights why an uncertain spinel assignment has a larger process penalty than an uncertain label for a readily soluble salt: the former often controls the refractory fraction and therefore drives pretreatment intensity and residual Zn.

12.2. Mineralogical mass balance

A robust mineralogical mass balance reconciles XRD-derived phase fractions with bulk elemental analysis and, where available, independently measured phase chemistry. For element i, closure can be reported as $C_i = [\Sigma_j(w_j x_{ij}) / X_i,bulk] \times 100$, where w_j is the mass fraction of phase j, x_{ij} is the concentration of element i in that phase, and $X_i,bulk$ is the independently measured bulk concentration. The uncertainty of phase fraction, phase chemistry, and bulk analysis should be propagated into the closure rather than reporting a single deterministic percentage. Zn and Fe

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are primary targets, but Mn and Cr are particularly informative for substituted spinels, Ca for acid-demand interpretation, Pb for volatilization or recovery, and Cl and S for soluble salts and process chemistry. Large closure errors can expose missing XRD-amorphous/unmodeled material, incorrect stoichiometric assumptions, unrecognized soluble phases, or biased QXRD. This approach prevents a nominal formula from silently controlling the elemental balance.

Table 6 condenses the phase-to-process translation that should guide critical interpretation.

Table 6. Metallurgical significance of validated EAFD phase assignments.

Validated phase/family	Likely process implication	Key caution
ZnO-rich fraction	Generally high direct leachability	Surface coatings/agglomeration can reduce accessibility
Zn-rich spinel/ferrite	Refractory Zn; may require roasting/reduction	Exact reactivity depends on substitution and defect chemistry
Magnetite/Fe-bearing spinels	Controls Fe redox and residue behavior	Maghemite/spinel overlap may bias interpretation
Ca-bearing phases	Acid consumption and secondary precipitation risk	Bulk Ca does not identify reactive mineral form
Chlorides/sulfates	Important for washing, corrosion, and purification	May be hydrated, deliquescent, or minor by XRD
Amorphous/disordered material	Potential unaccounted metals and reactivity	Requires standard-based quantification or independent balance

Note. Process implications are tendencies, not universal rules; particle association and operating conditions remain important.

12.3. Critical knowledge gaps

The literature reveals recurring gaps: excessive reliance on automated peak matching; limited use of quantitative Rietveld analysis in routine EAFD studies; inadequate measurement of XRD-amorphous/unmodeled material; over-specific assignment of cubic spinels as pure $ZnFe_2O_4$; insufficient validation with EPMA/WDS, Mössbauer, or XAS; sparse reporting of analytical uncertainty; weak standardization of sample preparation; limited treatment of microabsorption; few mineralogical mass balances; and incomplete integration between feed mineralogy, phase transformation, residue mineralogy, and metallurgical performance

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(Thottempudi et al., 2022; Toby, 2006; Trifunović et al., 2022; Tzvetanova et al., 2020; Wang et al., 2021; Wang et al., 2025; Westphal & Füllmann, 2012; Young, 2010; Zhang et al., 2024; Zhu et al., 2023).

12.4. Cross-study synthesis and evidentiary weighting

Across the EAFD literature, the first robust conclusion is heterogeneity rather than a universal phase inventory. Dust chemistry and mineralogy vary with steel grade, scrap composition, furnace practice, gas cooling, recirculation, and downstream conditioning. Consequently, phase labels and process responses should not be transferred uncritically between plants or campaigns, even when bulk Zn and Fe contents are similar (Abadi & Tang, 2024; Al-Harahsheh et al., 2019; Branca et al., 2020; Chen et al., 2011; Cherrat et al., 2025; De Buzin et al., 2017; He et al., 2026; Huang et al., 2024; Lin et al., 2017; Gargul et al., 2021; Matsukevich & Kulinich, 2022; Pereira & Fonseca, 2025; Rieger et al., 2021; Sajal & Ahmad, 2025; Thottempudi et al., 2022; Trifunović et al., 2022; Wang et al., 2021).

A second conclusion is that diffraction quality cannot be judged from peak visibility alone. Instrumental broadening, particle statistics, absorption contrast, fluorescence, preferred orientation, crystallite size, strain, and background modeling interact with reference-data quality and structural-model completeness. These effects are especially consequential for fine, Fe-rich, multiphase EAFD, where apparently small experimental biases can alter both detection and quantification of minor or overlapping phases (Balzar, 2011; Chipera et al., 2013; Cornell et al., 2013; Dvořák et al., 2017; Fan et al., 2026; He, 2018; Hill et al., 2010; Holder et al., 2019; Scardi & Leoni, 2010; Suetens & Guo, 2015; Zhang et al., 2024; Zhu et al., 2023).

The strongest recurring identification problem concerns cubic spinels. The literature supports treating $ZnFe_2O_4$, magnetite, maghemite, franklinite-type phases, and substituted Zn–Fe–Mn–Cr spinels as an evidentiary continuum rather than as labels that can always be separated by routine peak matching. When process interpretation depends on ferrite refractoriness, cation substitution, or Fe oxidation state, lattice parameters and search–match scores should be

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corroborated by phase chemistry or element-specific spectroscopy (Machitouen et al., 2026; Pickles, 2010; Tang et al., 2022; Wu et al., 2014).

Quantitative phase analysis improves the analytical value of XRD only when the refinement model is explicit about standards, amorphous or unmodeled material, microabsorption, preferred orientation, peak-shape assumptions, and model sensitivity. Round-robin, standards, and methodological studies consistently show that fit indices alone do not establish phase correctness or unbiased phase fractions. For EAFD, uncertainty should therefore be attached to the complete measurement-and-model chain, not only to least-squares standard errors (Aranda et al., 2012; Bish, 2013; Black et al., 2011; Christidis et al., 2020; Cline et al., 2015; Coelho, 2018; David, 2010; De la Torre et al., 2010; Dinnebier & Leineweber, 2018; Dinnebier & Billinge, 2008; Eberl, 2010; Gates-Rector et al., 2019; Gozzo, 2010; Gualtieri et al., 2019; Hammersley, 2016; Hiraga & Gomes, 2021; Itakura et al., 2025; Jansen et al., 2011; Kern & Madsen, 2012; Kleeberg & Monecke, 2010; Kniess et al., 2012; León-Reina et al., 2014; Lopresti et al., 2022; Lutterotti et al., 2010; Madsen & Scarlett, 2011; Madsen & Scarlett, 2012; Mittemeijer & Welzel, 2008; Pecharsky et al., 2015; Pernechele et al., 2025; Post et al., 2010; Prasianakis, 2024; Prescher et al., 2015; Rowles, 2021; Scarlett et al., 2018; Shen et al., 2025; Toby et al., 2013; Toby, 2006; Tzvetanova et al., 2020; Wang et al., 2025; Westphal & Füllmann, 2012; Whitfield, 2016; Young, 2010).

Automation can accelerate candidate generation, but the reviewed evidence does not justify replacing expert plausibility checks with an algorithmic phase label. Search–match or machine-learning output is strongest when it reports alternatives and uncertainty and is followed by orthogonal confirmation. SEM–EDS and automated mineralogy provide particle association, EPMA/WDS constrains phase chemistry, Mössbauer spectroscopy and X-ray absorption methods constrain Fe valence and coordination, and Raman spectroscopy can help resolve selected oxide polymorphs (Chateigner et al., 2012; Lee et al., 2020; Lee et al., 2021; Schuetzke et al., 2021; Szymanski et al., 2023; Fandrich et al., 2007; Fleet, 2013; Goldstein et al., 2018; Gottlieb et al., 2010; Gütlich & Bill, 2011; Nasdala et al., 2012; Orozco-Henao et al., 2022; Potts et al., 2011; Reed, 2011; Meena & Balraju, 2025; Wilke et al., 2011).

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Process studies further show that static feed mineralogy is insufficient when treatment deliberately changes phase assemblage. Reduction, calcination, microwave treatment, sulfation, chlorination, and related thermal routes can alter Zn-bearing spinels and Fe oxides through reduction, oxidation, volatilization, and solid-state reaction. Mechanistic interpretation is strongest when phase evolution is measured across feed, intermediate, and product or residue states and reconciled with elemental balances (Ahmad et al., 2022; Anzulevich et al., 2021; Brandner & Antrekowitsch, 2021; Chang et al., 2022; Chen et al., 2023; Fan et al., 2026; Grudinsky et al., 2022; Hamann et al., 2024; Ju et al., 2025; Li et al., 2023; Ma et al., 2022; Omran & Fabritius, 2019; Qiu et al., 2026; Wu et al., 2023; Xiong et al., 2024; Xue et al., 2025; Yang et al., 2024; Zhang et al., 2014).

The same principle applies to hydrometallurgy. Extraction data become mineralogically interpretable only when they are paired with feed and residue characterization, because soluble Zn phases, refractory spinels, secondary precipitates, and particle-scale accessibility can contribute simultaneously to an observed recovery. The most defensible phase-to-process claims therefore combine QXRD, independent phase chemistry, solution and residue analyses, and mass-balance closure rather than inferring reactivity from a nominal mineral label alone (Bakkar, 2014; Binnemans & Jones, 2020; Fan et al., 2026; Gamutan et al., 2024; Halli et al., 2018; Halli et al., 2017; Kalpakli et al., 2021; Kinnunen et al., 2025; Kukurugya & Vindt, 2015; Kul et al., 2015; Lanzerstorfer et al., 2020; Li & Wang, 2010; Miranda & Iglesias-González, 2026; Montenegro et al., 2016; Mousavi & Mousavi, 2024; Oustadakis et al., 2010; Palimąka et al., 2018; Pereira & Fonseca, 2026; Pereira & Santos, 2025a; Pereira & Santos, 2025b; Prasetyo et al., 2020; Teimouri et al., 2022; Terrones-Saeta et al., 2021; Toporkova & Prodanova, 2020; Trifunović et al., 2024; Yang et al., 2025; Zoraga & Ilhan, 2020; Zoraga et al., 2022).

13. Conclusions and Outlook

XRD is indispensable for EAFD characterization, but phase identification should not be equated with phase confirmation. The central limitation is not the ability of diffraction to detect crystalline order; it is the tendency to assign chemically specific names to structures that may be

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compositionally substituted, overlapped, nanocrystalline, structurally disordered, or partly unmodeled. For Zn-bearing spinels in particular, a peak set compatible with $ZnFe_2O_4$ does not demonstrate a pure stoichiometric ferrite, and an unqualified franklinite assignment is not justified without independent evidence for the relevant phase chemistry. Quantitative Rietveld analysis can substantially improve the mineralogical description, but only when the XRD-amorphous/unmodeled fraction, microabsorption, preferred orientation, model completeness, between-preparation variability, and model-choice sensitivity are addressed transparently. Orthogonal evidence from microanalysis and spectroscopy should be targeted to the ambiguity that matters for the specific process decision, recognizing that complementary methods reduce rather than abolish uncertainty. The most defensible workflow therefore progresses from representative sampling, documented sample preservation, and controlled acquisition to cautious phase-family identification, uncertainty-aware QXRD, hypothesis-driven multimethod validation, and elemental mineralogical mass-balance closure. Future work should prioritize standardized EAFD reference materials, interlaboratory comparisons, explicit reporting of practical detection limits and uncertainty, better structural models for substituted industrial spinels, nonambient phase-transformation measurements, and paired feed–intermediate–residue datasets. The quality of metallurgical interpretation cannot exceed the quality of the phase identification on which that interpretation is based.

Declarations

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Conflict of Interest

The author declares no conflict of interest.

Data Availability

No new experimental datasets were generated. All quantitative values discussed in the review are traceable to the cited literature.

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Author Contributions

The author performed conceptualization, critical analysis, literature synthesis, visualization, and manuscript preparation.

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