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Recovery of Zirconium from Primary and Secondary Resources: Chemistry, Zr/Hf Separation, Electrochemical Routes, and Industrial Perspectives

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Abstract

Zirconium is a strategically important, oxide-forming metal whose production chain spans refractory mineral decomposition, the difficult separation of zirconium/hafnium, halide chemistry, and high-purity metal production. This structured critical narrative review evaluates a core corpus of 111 peer-reviewed journal publications from 2020 through August 2026, supplemented by explicitly identified normative and foundational sources used for industrial specification and process context. The retained evidence base is auditable from the final 111-paper corpus onward; however, original database hit counts, duplicate-removal records, and article-by-article exclusion logs were not preserved, so the discovery stage cannot be independently reconstructed and the review is not presented as PRISMA-compliant. The evidence is organized around feedstock reactivity, aqueous and molten-salt speciation, industrial decomposition and chlorination, Zr/Hf separation, electrochemical conversion, secondary-resource recycling, and environmental-economic integration. Studies are interpreted using an evidence-maturity rubric that ranges from mechanistic or theoretical evidence to industrial or standardized practice. The synthesis shows that zircon decomposition is technically mature but reagent- and energy-intensive, whereas Zr/Hf and trace-impurity separation often controls access to nuclear-grade products. TBP/nitric acid extraction remains a process benchmark, while phosphinic and phosphate extractants, extraction resins, ion-imprinted membranes, and porous adsorbents broaden the selectivity toolkit but remain limited by regeneration, capacity, durability, and evidence for multicycle performance. Molten-salt electrodeposition and consumable-anode concepts can integrate conversion and metal production, yet salt purity,

JIRRS
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Pages: 1-58

current efficiency, electrode compatibility, and product harvesting remain constraints for scale-up. Secondary routes indicate that direct alloy recycling, selective cladding treatment, and functional upcycling should be selected based on feed quality and product specifications. For high-value products, zirconium recovery must be reported together with product form, Hf content, interstitial impurities, residual halogens, and the applicable specification. The review therefore identifies hybrid flowsheets, quantitative product qualification, and closed reagent or salt loops as the most credible directions for zirconium metallurgy.

Keywords: zirconium recovery; zircon processing; zirconium–hafnium separation; solvent extraction; molten-salt electrolysis; secondary resources.

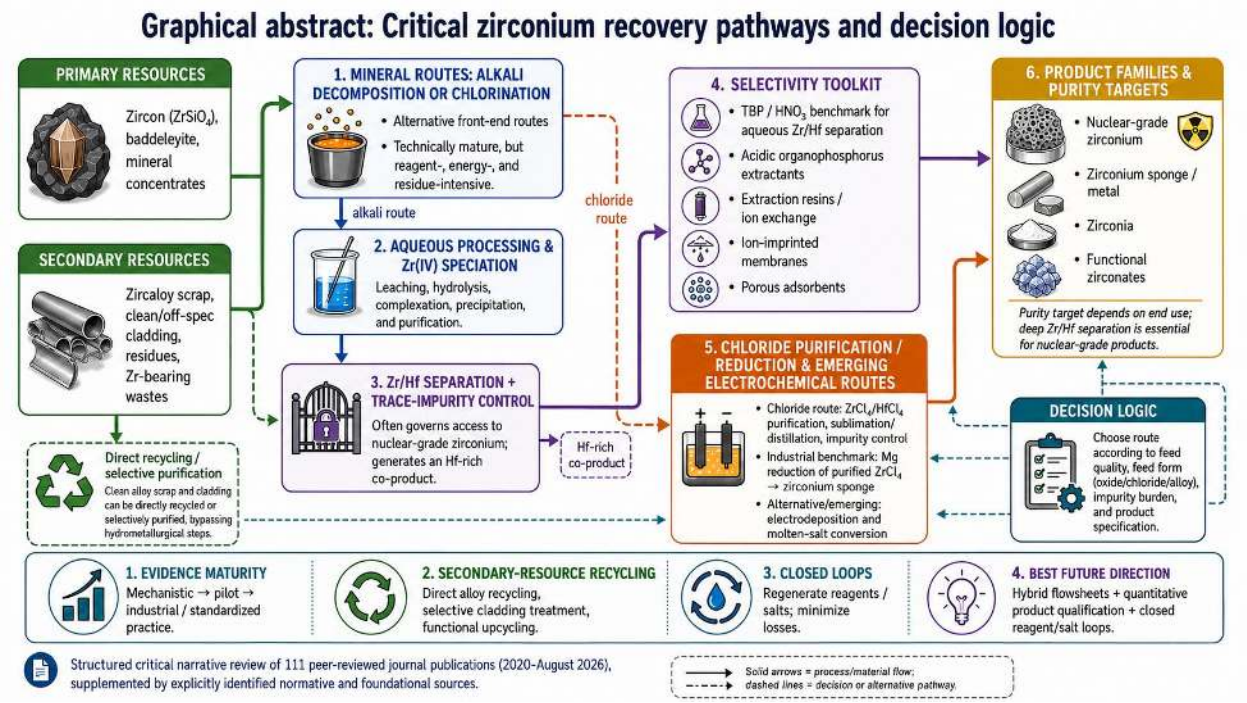
Highlights

- Zirconium recovery is governed by coupled mineral decomposition, speciation, Zr/Hf separation, and final purity rather than by extraction yield alone.
- Conventional alkali/chloride routes remain industrially credible, while low-temperature chlorination and molten-salt electrochemistry target lower process intensity and improved circularity.
- Recent Zr/Hf separation research is shifting from bulk solvent extraction toward ligand-designed resins, membranes, porous adsorbents, and hybrid molecular-separation systems.
- Secondary zirconium from alloy scrap, nuclear cladding, and process residues can materially improve circularity, but impurity control, salt/reagent closure, and product qualification determine scale-up.

JIRRS
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Pages: 1-58

Graphical abstract



1. Introduction

Zirconium occupies an unusual position in extractive metallurgy because the same element supports high-volume ceramic and refractory uses and high-specification nuclear applications. Zircon ($ZrSiO_4$) remains the dominant mineral source, commonly recovered as a co-product of heavy-mineral-sand operations, whereas baddeleyite and several industrial or nuclear secondary streams provide more feed-specific opportunities. Recent material-flow and resource studies emphasize that zirconium supply must be interpreted together with heavy-mineral grades, geographic concentration, co-product economics, environmental reporting, and the rapidly growing anthropogenic stock of zirconium-containing products (Perks & Mudd, 2021, 2022; Perks et al., 2022; Zhu et al., 2023; Liu et al., 2026).

The technical difficulty is not scarcity alone. Zircon is chemically robust, ZrO_2 is highly stable, Zr(IV) hydrolyzes strongly in water, and zirconium follows hafnium through most primary concentration and chemical conversion steps because the two elements have nearly identical ionic radii and closely related coordination chemistry. As a consequence, the production

JIRRS
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Pages: 1-58

of ordinary zirconia and the production of nuclear-grade zirconium are different process problems: the former is dominated by efficient mineral opening and impurity removal, whereas the latter adds a stringent Zr/Hf separation and trace-contaminant requirement. Recent solution-chemistry and speciation studies therefore move the field away from a simple "leach–extract–precipitate" narrative toward explicit control of hydrolysis, anionic complex formation, solvation, adsorption, and phase transfer (Blumenthal, 1958; Bertoli et al., 2021; Dong et al., 2025; Zhang et al., 2024; Zouari et al., 2021).

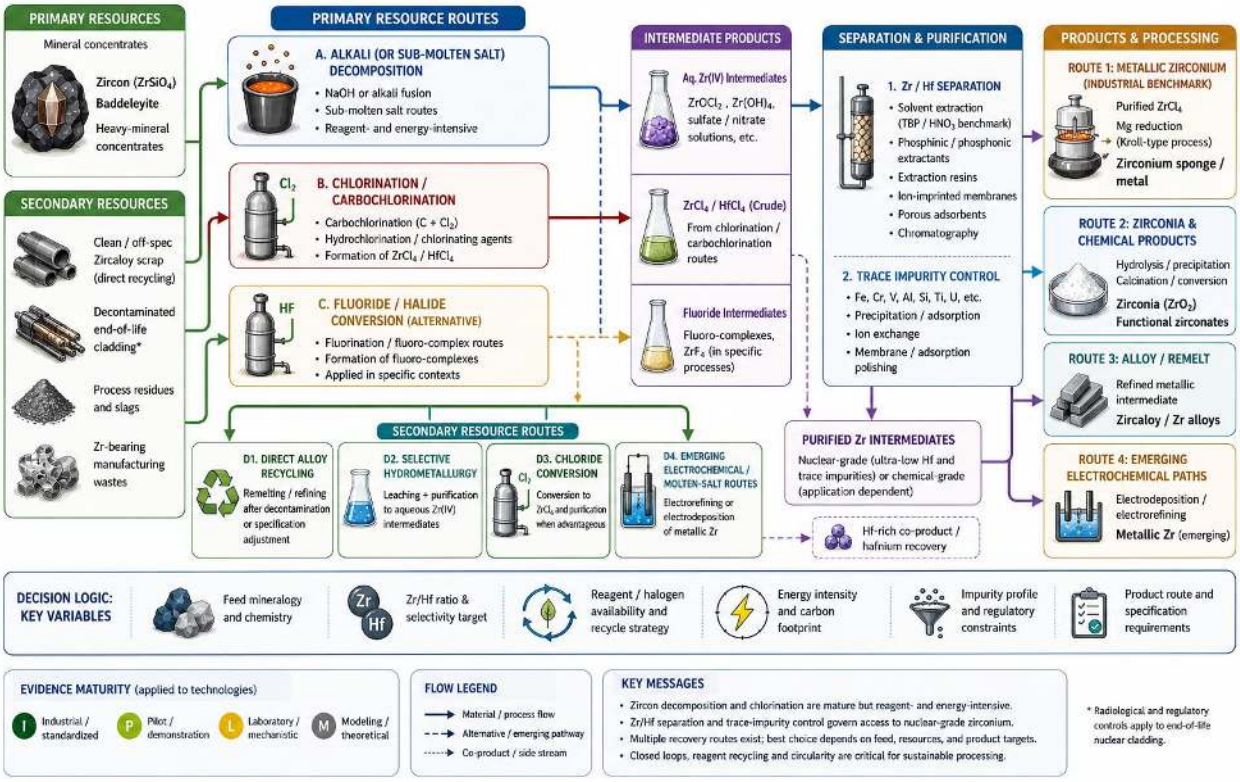
Industrial zirconium production still relies on chemically intensive steps. Alkali decomposition converts the refractory silicate framework to soluble or leachable sodium silicates and zirconates, while chloride-based routes create volatile $ZrCl_4$ suitable for purification and subsequent reduction. In parallel, the 2020–2026 literature has expanded three alternative directions: (i) selective aqueous separations using new extractants, resins, membranes, and porous materials; (ii) direct or semi-direct electrochemical reduction in chloride and fluoride melts; and (iii) recycling of zirconium alloys and nuclear cladding using remelting or low-temperature chemical decladding (S. Li et al., 2021a; Vestal et al., 2023; Vutova et al., 2025; Zhou et al., 2026).

Figure 1 summarizes the integrated process landscape used in this review. The figure is deliberately feed-to-product rather than technology-by-technology because the critical engineering question is where each route creates selectivity, where it transfers impurities, and whether its reagent and salt circuits can be closed.

JIRRS
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Figure 1. *Integrated zirconium recovery landscape*



Note. Author-developed synthesis adapted from Perks and Mudd (2021, 2022), Song et al. (2020), S. Li et al. (2021a), Vestal et al. (2023), and Zhou et al. (2026). The internal evidence-maturity key is a simplified visual grouping of the E1–E5 rubric defined in Section 2, not a separate readiness scale.

As Figure 1 indicates, no route can be judged solely by zirconium extraction. Mineral opening, intermediate selection, Zr/Hf purification, and final product form are interdependent. A low-temperature or high-yield front end may be unattractive if it produces a dilute stream that requires many extraction stages, whereas a high-temperature route may remain competitive if volatile intermediates facilitate high-purity separation and recycling.

Accordingly, the objective of this review is to critically evaluate zirconium recovery routes reported from 2020 through August 2026 from primary and secondary sources, with emphasis on chemical principles, Zr/Hf selectivity, evidence maturity, process integration, and industrial feasibility. The review asks four questions: where is selectivity generated; what reagents or energy carriers must be recycled for the route to be credible; which impurities control product qualification; and which emerging approaches offer a realistic pathway beyond laboratory-scale demonstrations?

JIRRS
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The review is organized around a Feed–Speciation–Selectivity–Conversion–Qualification (FSSCQ) Framework. The framework treats route interfaces—mineral opening → bulk impurity removal → Zr/Hf separation → conversion to a controlled intermediate or metal → product qualification—as coupled decisions, with reagent, salt, water, and energy-loop closure as cross-cutting constraints. It therefore evaluates not only individual technologies but also whether adjacent unit operations are chemically and operationally compatible. This interface logic foregrounds hybrid flowsheets: mature front-end chemistry may be combined with targeted molecular separation, polishing, membrane enrichment, or electrochemical finishing when the combined train better satisfies product specifications and loop-closure requirements.

2. Review Scope and Critical Method

2.1 Corpus construction and search architecture

The analytical corpus comprises 111 peer-reviewed journal publications published from 2020 through August 2026. The final search update and corpus cutoff used for this revision was 31 August 2026. During revision, the search architecture was reconstructed and audited to improve transparency without retrospectively claiming PRISMA compliance. Scopus, Web of Science, and ScienceDirect were treated as the principal bibliographic environments, and the search logic was organized into five complementary blocks covering general zirconium recovery, Zr/Hf separation, molten-salt electrochemistry, secondary-resource recycling, and sustainability. Because the original database-level hit counts, duplicate-removal records, and article-by-article exclusion log were not preserved, these quantities were not recreated from inference. The manuscript is therefore retained as a structured critical narrative review rather than re-labeled as a systematic review.

The reconstructed Boolean architecture was: (i) core recovery: (zirconium OR zircon OR ZrO2 OR ZrCl4 OR Zircaloy) AND (recovery OR extraction OR recycling OR processing OR hydrometallurg* OR pyrometallurg* OR chlorination OR "solvent extraction" OR "ion exchange" OR electrochem* OR "molten salt*"); (ii) Zr/Hf separation: (zirconium OR "Zr(IV)" OR ZrCl4) AND (hafnium OR "Zr/Hf") AND ("solvent extraction" OR resin* OR chromatograph* OR membrane* OR adsorb* OR "ionic liquid*" OR "deep eutectic solvent*");

JIRRS
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(iii) electrochemistry: (zirconium OR ZrCl4 OR ZrO2) AND ("molten salt*" OR electrolysis OR electrodeposition OR electrorefining OR electroreduction); (iv) secondary resources: (zirconium OR Zircaloy OR cladding) AND (recycling OR remelting OR hydrochlorination OR decladding OR secondary); and (v) sustainability: zirconium AND ("life cycle" OR LCA OR techno-economic OR circularity OR "material flow"). Search terms were adapted to database syntax without changing the underlying concepts. Database-specific syntax was adapted without changing the concepts: Scopus searches used TITLE-ABS-KEY fields and Scopus wildcards where supported; Web of Science used Topic (TS=) logic; and ScienceDirect terms were adapted to its available title/abstract/keyword or full-text search fields. Early-online 2026 records were eligible only when stable journal metadata, a traceable DOI or permanent publisher record, and full text suitable for critical appraisal were available by the cutoff. All 14 retained 2026 journal records in the analytical corpus carry DOI traceability in the reference list.

Normative and foundational sources were retained separately from the 111-paper analytical corpus to connect recent innovation to durable principles of zirconium chemistry, Zr/Hf separation, solvent extraction, Kroll metallurgy, molten-salt thermodynamics, nuclear-product specification, and radiological safety (ASTM International, 2021a, 2021b; Banda & Lee, 2015; Blumenthal, 1958; International Atomic Energy Agency, 2014; Janz, 1967; Rydberg, 2004; Stephens, 1984; Xu et al., 2015). These sources are not counted in the 111-paper analytical corpus and are not used to inflate the 2020–2026 evidence base.

2.2 Eligibility, evidence directness, and maturity coding

Eligible core papers were peer-reviewed journal articles within the stated publication window that provided either direct zirconium evidence or transferable process evidence with a clearly identified relevance to zirconium flowsheets. Direct evidence includes zircon decomposition, Zr/Hf separation, zirconium electrochemistry, recycling of zirconium alloys or cladding, and zirconium-specific life-cycle or material-flow studies. Transferable evidence includes process concepts such as impurity precipitation, solvent regeneration, or electrorefiner operation when the study did not itself demonstrate zirconium recovery; such evidence is explicitly labelled and is not used to support zirconium-specific performance claims. Duplicate

JIRRS
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records, non-peer-reviewed items, and sources outside the time window were excluded from the analytical corpus unless retained separately as normative or foundational context.

Evidence maturity was coded on an ordinal E1–E5 rubric: E1, mechanistic, thermodynamic, computational, or analytical evidence without an integrated recovery demonstration; E2, laboratory proof-of-concept or batch separation/electrodeposition; E3, multicycle, continuous, integrated bench-scale, or flowsheet-oriented demonstration; E4, pilot or engineering-scale evidence, or a strongly validated transferable unit operation under relevant process conditions; and E5, established industrial practice or standardized production/product qualification. The levels describe evidence maturity rather than intrinsic scientific quality and are used to prevent an exceptional batch separation factor from being interpreted as industrial readiness. Coding was performed by the single author/reviewer of the corpus. No independent second-coder exercise or reliability coefficient was available for this revision; therefore, E1–E5 assignments are explicitly treated as single-reviewer interpretive maturity judgments rather than reliability-tested classifications. No Cohen's kappa, Krippendorff's alpha, or percentage-agreement statistic is claimed. Supplementary Table S1 exposes the source-level coding and includes a stratified verification worksheet so that an independent coder can be added if required by the editor.

2.3 Evidence maturity, methodological quality, and industrial transferability

Three distinct appraisal dimensions are used and should not be conflated. Evidence maturity describes the degree of process integration and scale represented by E1–E5; it does not score the intrinsic scientific rigor of a study. Methodological quality refers to whether the study design, measurements, balances, controls, and interpretation are adequate for the question being asked; a rigorous mechanistic or spectroscopic study may therefore remain E1 while being methodologically strong. Industrial transferability describes whether evidence is direct to zirconium or instead supports a unit-operation principle that may transfer to a zirconium flowsheet. Supplementary Table S1 records these dimensions separately and adds a reporting-completeness field based on five process-relevant domains: quantitative performance,

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operability/scale, regeneration or recycle, impurity tolerance, and product qualification. Reporting completeness is a transparency descriptor, not a quality score.

2.4 Critical comparison criteria and limitations

The critical comparison uses six recurring criteria: (1) feedstock conditioning and mineral opening; (2) zirconium recovery and concentration; (3) Zr/Hf and trace-impurity selectivity; (4) reagent, salt, and water recycle; (5) energy and residue burden; and (6) evidence maturity. Where available, quantitative extraction, separation-factor, loading, recovery, current-density, and product-composition data are reported together with the corresponding evidence level. Missing operability variables—such as stripping, regeneration, phase disengagement, current efficiency, electrode life, multicycle stability, or product harvesting—are treated as unresolved rather than inferred.

Benchmarking note. Comparative figures and tables provide conditional process benchmarking, not definitive technology ranking. Route selection depends on feed composition, target product, impurity tolerance, regulatory context, reagent and salt closure, available infrastructure, and site-specific mass and energy balances. Figures 1–5 are author-developed conceptual syntheses rather than outputs of statistical modeling. In particular, Figure 5 is an ordinal maturity–circularity positioning map; it is not an LCA result, formal TRL score, or quantitative ranking.

The principal methodological limitation is therefore not hidden: the review can audit the scope, eligibility logic, evidence directness, and critical-assessment framework, but it cannot reconstruct unavailable historical database hit counts or an original PRISMA flow record. The resulting synthesis should be interpreted as a transparent, structured critical narrative review of a curated recent corpus, strengthened by quantitative cross-comparison where the underlying papers report comparable metrics.

3. Resource Base, Supply Chain, and Feedstock Classes

3.1 Primary zirconium resources and heavy-mineral context

Primary zirconium supply is strongly coupled to the mining and separation of heavy-mineral sands. Global assessments show that zircon production data, grades, reserves, and sustainability performance vary substantially across deposits and reporting systems, and that

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DOI: 10.66104/s3mdb246
Pages: 1-58

declining ore grades can shift energy and material burdens upstream even when downstream chemical recovery is unchanged (Perks & Mudd, 2021, 2022; Perks et al., 2022). Recent life-cycle work on valuable heavy-mineral concentration further demonstrates that the environmental footprint of zirconium feed cannot be separated from the mining and physical-separation sequence that produces the concentrate (Laghaei et al., 2025, 2026). Morfino et al. (2022) similarly show that application-level comparisons involving zircon sand can be sensitive to the upstream life-cycle inventory used for the mineral.

3.2 Secondary sources and the expanding anthropogenic stock

Secondary zirconium resources differ sharply in composition and risk. Clean manufacturing scrap is already alloyed and may be suitable for remelting; irradiated or potentially contaminated cladding requires decontamination and radionuclide-aware flowsheets; hydrometallurgical or uranium-processing residues may contain zirconium together with strongly interfering elements; and lower-grade zirconium wastes may be more valuable as precursors for zirconates than as sources of nuclear-grade metal. Dynamic material-flow work for China and global-to-national zirconium-cycle analysis indicate that anthropogenic stocks are becoming large enough to justify dedicated recovery pathways rather than treating zirconium as an incidental residue component (Zhu et al., 2023; Liu et al., 2026).

Table 1 classifies the principal feed types and their process implications. The distinction is important because the same recovery technology can perform very differently when the feed changes from a clean zircon concentrate to oxidized nuclear cladding or a multicomponent industrial residue.

Table 1. *Primary and Secondary Zirconium Feedstocks and Their Processing Implications*

Feed class	Chemical context	Processing objective	Representative route	Critical constraint
Zircon concentrate	$ZrSiO_4$; Hf follows Zr; Si-rich matrix	Break refractory silicate lattice	NaOH fusion/sub-molten salt; chlorination; fluorination	Silica/alkali balance; Zr/Hf separation downstream
Baddeleyite / ZrO_2-rich mineral	ZrO_2 with gangue	Activate/reduce stable oxide	Chlorination; molten-salt reduction; acid routes after activation	High oxide stability; impurity dissolution

JIRRS
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Feed class	Chemical context	Processing objective	Representative route	Critical constraint
Clean alloy scrap	Zr alloys with Sn/Nb/Fe/Cr depending on alloy	Preserve metal value	Vacuum induction/electron-beam remelting; refining	Interstitial O/N/H; alloying-element buildup
Nuclear cladding	Zircaloy/ZIRLO; oxide layer; contamination risk	Declad and decontaminate selectively	Hydrochlorination; sulfur-chloride route; electrorefining	Radioactive contaminants; oxide scale; product requalification
Process/uranium residues	Zr with U, Fe, Al, Sc, salts and oxides	Release Zr selectively from complex matrix	Alkali decomposition + acid leach; solvent extraction/adsorption	Co-dissolution; residue classification; reagent consumption
Zr-bearing waste for functional products	Variable Zr content	Convert directly to value-added zirconate	Na_2ZrO_3 synthesis / carbonation-linked concepts	Product market size; feed impurity tolerance

Note. Author-developed synthesis adapted from Perks and Mudd (2021, 2022), Fan et al. (2023), Pereira et al. (2020), Vestal et al. (2023), Fu et al. (2025), and Gamba et al. (2026).

Table 1 shows why secondary-resource recovery cannot be assessed by zirconium grade alone. Clean scrap may bypass mineral decomposition completely, whereas cladding demands selective decontamination and high assurance. Conversely, a chemically heterogeneous waste may be better directed to a zirconate or ceramic product if nuclear-grade purification would destroy the circularity advantage.

4. Chemistry and Thermodynamic Constraints

4.1 Stability of ZrO_2 and $ZrSiO_4$

The core thermodynamic obstacle is zirconium's strong oxygen affinity. ZrO_2 is highly stable, while zircon incorporates zirconium into a silicate lattice that resists ordinary mineral-acid attack. Electrochemical equilibrium analysis of the zirconium–water system reinforces the narrow conditions under which metallic zirconium is stable in aqueous environments and explains why direct aqueous electrowinning is not a realistic analogue of copper or zinc electrowinning (Jin et al., 2020). Solution and surface thermodynamic work on zirconia further shows that even trace aqueous behavior depends on polymorph, surface state, and chemical

JIRRS
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Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

environment (Zouari et al., 2021). Thermodynamic assessments of Zr–B–C–O and fluoride-containing systems are therefore directly relevant to high-temperature reduction and molten-salt process design (Pham et al., 2023; Dumaire et al., 2024).

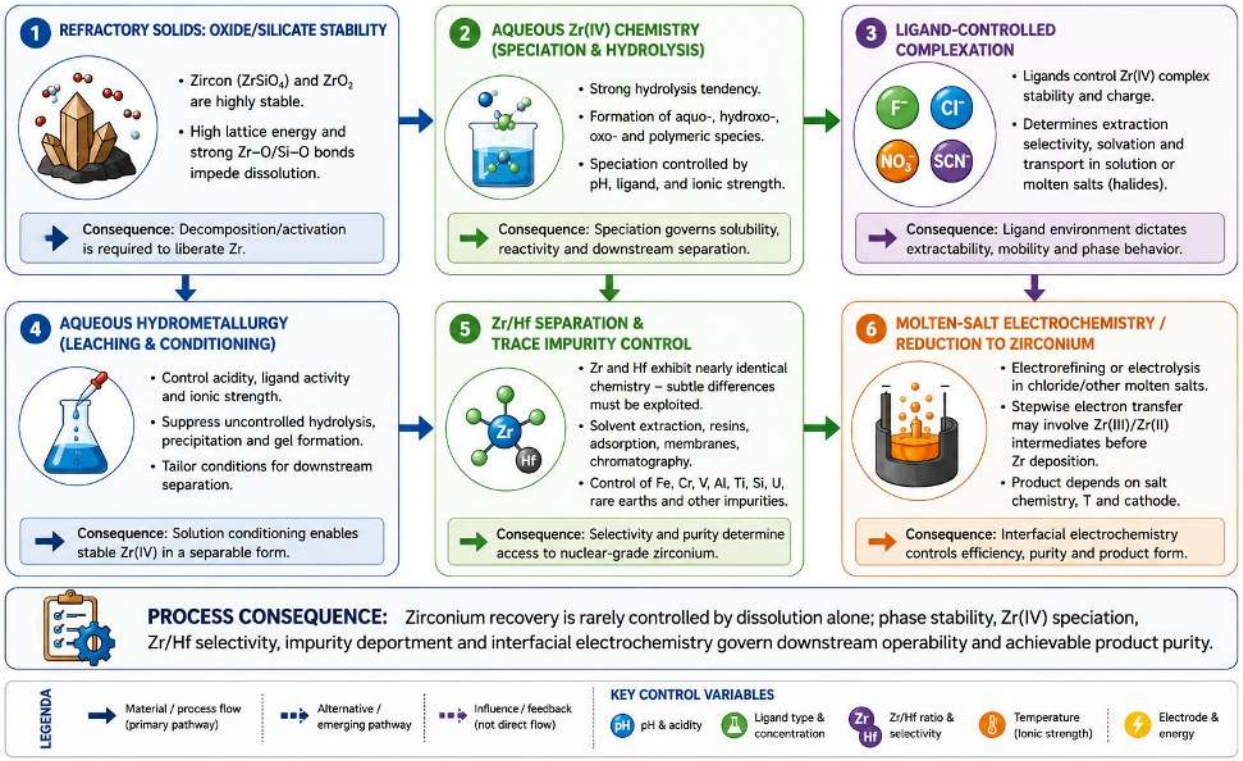
4.2 Aqueous speciation: hydrolysis versus complexation

In aqueous solution, Zr(IV) is a hard, highly charged cation with strong hydrolysis and polymerization tendencies. Industrially useful separation is made possible by shifting zirconium into nitrate, chloride, fluoride, sulfate, thiocyanate, or organophosphorus-associated complexes. Bertoli et al. (2021) demonstrated the complexity of Zr/Hf speciation in fluoride–nitric media using mass spectrometry and DFT, while Zhang et al. (2024) linked denitration-driven changes in aqueous zirconium forms to extraction behavior. Novikov et al. (2025) further illustrate that Zr(IV) forms chemically meaningful complexes even with oxyanions such as Tc(VII) and Re(VII) in liquid–liquid systems, emphasizing that minor ligands can alter phase transfer and should not be ignored in nuclear or residue-derived liquors.

Figure 2 organizes these chemical constraints into the three process environments most relevant to current research. The figure links refractory oxide/silicate stability, ligand-controlled aqueous speciation, and multivalent molten-salt electrochemistry.

JIRRS
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Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Figure 2. *Chemistry and speciation constraints governing zirconium processing*



Note. Author-developed synthesis adapted from Jin et al. (2020), Bertoli et al. (2021), Zouari et al. (2021), Dumaire et al. (2024), and Zhang et al. (2024).

Figure 2 highlights a recurring design principle: zirconium recovery is a species-management problem. In hydrometallurgy, too little complexation promotes hydrolysis and precipitation; too much complexation can suppress stripping or reduce Zr/Hf discrimination. In molten salts, the activities of chloride and fluoride change the reduction pathways and nucleation behavior. The practical optimum is therefore not the condition with the strongest zirconium binding, but the condition that creates a controllable sequence of binding, transfer, release, and product formation.

4.3 Implication for Zr/Hf separation

Because Zr(IV) and Hf(IV) share charge, coordination preferences, and closely similar radii, separations depend on small free-energy differences multiplied over repeated equilibrium stages. This is why solvent extraction, chromatography, adsorption, membranes, and chloride distillation remain central. Recent mechanistic studies using dibenzoylmethane, phosphate esters,

JIRRS
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Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

phosphinic acids, and functionalized porous materials show that selectivity arises through subtle differences in ligand geometry, hydration, steric environment, and complex stability rather than through a single universal chemical discriminator (Luo et al., 2024; Qi et al., 2026; Wang, Liu, Xu, et al., 2023; Wan et al., 2026).

5. Industrial Decomposition and Chloride-Based Recovery Routes

5.1 Alkali decomposition of zircon

Alkali decomposition remains one of the most intelligible routes for converting zircon into water- and acid-processable intermediates. Song et al. (2020) reported a two-step NaOH fusion/water-leaching scheme in which zircon decomposition reached 97.25% under optimized staged fusion conditions at 700 °C. The staged design limits formation of less desirable mixed sodium zirconium silicates and separates sodium silicate from sodium zirconate intermediates. Subuki et al. (2020) likewise examined caustic fusion for zirconia powder production, confirming the continued relevance of alkali activation for zircon concentrates.

More recent sub-molten-salt work seeks lower-temperature control. Sun et al. (2024) showed that Na_2ZrSiO_5 formation can impair Zr/Si separation during NaOH treatment and identified conditions that favor Na_2ZrO_3 and Na_2SiO_3 formation. This is an important advance because it reframes alkali consumption: excess caustic is not merely a reagent-cost issue; it changes the phase pathway and therefore downstream washing, silica separation, and liquor composition. Fan et al. (2023) extended alkali decomposition to hazardous uranium purification waste, coupling it with acid leaching to release uranium and zirconium from a complex secondary matrix.

5.2 Chlorination, hydrochlorination, and $ZrCl_4$ purification

The chloride route remains strategically important because $ZrCl_4$ is volatile, chemically versatile, and compatible with reduction to metal. Borjas Nevarez et al. (2021) investigated recovery of zirconium from Zircaloys by hydrochlorination, while Balaji Rao, Gupta, et al. (2024) examined chemical characterization across zirconium sponge process intermediates. Trace-halogen control also matters for product qualification; Balaji Rao, Bano, et al. (2025) developed a pyrohydrolysis–ion chromatographic approach for chlorine determination in PHWR

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structural materials, illustrating the analytical rigor required when chloride chemistry is used upstream.

A notable 2023 development is the use of low-temperature sulfur-chloride chemistry for cladding. Vestal et al. (2023) demonstrated selective chlorination of unirradiated zirconium-alloy cladding using S_2Cl_2 at approximately 348–373 K, followed by dissolution and recrystallization of $ZrCl_4$ in $SOCl_2$. The work was demonstrated on 3–50 g scales with complete chlorination in roughly 3–4 h, and the presence of depleted UO_2 pellets did not prevent the reaction. Conrad et al. (2023) evaluated radiolytic behavior of sulfur chlorides relevant to this concept, providing essential evidence for reagent stability in nuclear applications. Mondal and Mayes (2026) place such cladding-extraction approaches within the broader evolution of nuclear fuel-cycle head-end processing.

5.3 Distillation and final chloride separation

For nuclear-grade zirconium, chloride formation alone does not solve the Hf problem. Ma et al. (2025) analyzed molten-salt distillation of $ZrCl_4/HfCl_4$ and reported a representative relative volatility of about 1.18 in $KAlCl_4$-containing melts over 623–773 K at the studied composition. That modest difference in volatility confirms both the feasibility and the stage-intensity of the separation. The route is attractive because it operates in a chloride-based process family that can integrate with $ZrCl_4$ production, but its industrial value depends on corrosion-resistant containment, vapor handling, salt purification, and closed chloride management.

5.4 Kroll reduction, sponge production, and product qualification

For metallic zirconium, purified and low-hafnium $ZrCl_4$ is the conventional bridge between chemical purification and metal production. In the Kroll route, zirconium tetrachloride is reduced with magnesium ($ZrCl_4 + 2Mg \rightarrow Zr + 2MgCl_2$) under controlled inert or vacuum-compatible conditions. Residual magnesium and $MgCl_2$ are then removed, typically by high-temperature vacuum separation, leaving a porous zirconium sponge that is crushed, blended, sampled, and qualified before melting or alloy production. The process remains the industrial reference because it decouples volatile-chloride purification from metal reduction and protects

JIRRS
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zirconium from oxygen and nitrogen contamination (Stephens, 1984; Xu et al., 2015; Balaji Rao, Gupta, et al., 2024; ASTM International, 2021b).

The Kroll step does not, by itself, solve the Zr/Hf problem. If $HfCl_4$ accompanies $ZrCl_4$ into the reduction reactor, hafnium is transferred into the sponge; consequently, the feasibility of nuclear-grade material depends on upstream Zr/Hf separation and final analytical qualification. ASTM B349/B349M designates Reactor Grade R60001 for nuclear sponge and identifies low hafnium as a defining characteristic of the grade, while downstream wrought-product standards impose additional interstitial and metallic-impurity controls (ASTM International, 2021a, 2021b). This distinction is critical when comparing emerging electrochemical routes with the conventional chain: an alternative process must replace not only the reduction reaction but also the impurity-control and product-qualification functions embedded in the chloride–Kroll flowsheet.

From a process-integration perspective, the key liabilities are chloride inventory, magnesium consumption and recycle, vacuum duty, corrosion-resistant equipment, and the handling of MgCl2-rich by-products. Conversely, the route's strength is that each major chemical transition produces a concentrated intermediate that can be sampled, purified, and controlled. Thus, lower-temperature or electrochemical alternatives should be benchmarked against the complete functional sequence—purification, reduction, separation, sponge recovery, and qualification—rather than against the Mg-reduction reaction alone.

Table 2 compares the principal decomposition and chloride routes and makes explicit where each route transfers rather than eliminates the separation burden.

Table 2. *Representative Industrial and Near-Industrial Zirconium Decomposition and Chloride Routes*

Route	Feed/window	Representative outcome	Critical assessment	Evidence
Two-step NaOH fusion + water leach	Zircon; ~700 °C staged fusion	97.25% zircon decomposition reported	Mature chemistry; high alkali/thermal duty; sodium-silicate management	Song et al. (2020)

JIRRS
Journal International Review of Research Studies

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Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Route	Feed/window	Representative outcome	Critical assessment	Evidence
NaOH sub-molten salt	Zircon; ~245 °C optimized study condition	Controls Na_2ZrSiO_5 and separates Zr/Si as sodium phases	Lower temperature, but high caustic inventory and long contact time	Sun et al. (2024)
Caustic fusion to zirconia	Zircon sand; high-temperature activation	Zirconia precursor production	Simple chemistry; washing and alkali recovery determine footprint	Subuki et al. (2020)
Hydrochlorination	Zircaloy; chloride conversion	Zr recovered as chloride species	Potentially direct for scrap; corrosion and gas handling remain central	Borjas Nevarez et al. (2021)
Low-T $S_2Cl_2/SOCl_2$ chlorination	Zr-alloy cladding; 348–373 K	3–50 g demonstration; ~3–4 h complete chlorination	Low-temperature selectivity; radiolytic validation and solvent recycle required	Vestal et al. (2023); Conrad et al. (2023)
Molten-salt $ZrCl_4/HfCl_4$ distillation	Purified chloride; 623–773 K data basis	Relative volatility ~1.18 at representative composition	Nuclear-grade relevance; stage count, corrosion and salt control are critical	Ma et al. (2025)
Kroll Mg reduction + vacuum separation	Purified, low-Hf $ZrCl_4$; Mg reductant	Zr sponge after $Mg/MgCl_2$ removal	Industrial reference for metal; Hf must already be controlled; vacuum, Mg/Cl recycle, and interstitial control remain decisive	Xu et al. (2015); Balaji Rao, Gupta, et al. (2024); ASTM International (2021b)

Note. Author-developed synthesis. Numerical values are representative literature conditions, not universal design values.

Table 2 demonstrates that conventional routes remain competitive because they create concentrated, chemically well-defined intermediates. Their weakness is not necessarily recovery, but loop closure: caustic must be regenerated or economically managed; chloride reagents and salts must be recycled; and energy/corrosion costs must be balanced against downstream simplification. This explains why emerging research often modifies a conventional chemistry rather than abandoning it completely.

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

6. Hydrometallurgical Recovery and Zr/Hf Separation

6.1 Leaching, precipitation, and purification before Zr/Hf separation

Hydrometallurgical zirconium processing usually begins after the refractory zircon lattice has already been opened. The resulting liquor chemistry controls whether zirconium can be concentrated cleanly or whether downstream extraction is burdened by silica, iron, uranium, scandium, and other metals. Ali (2022) demonstrated purification of zirconium oxide from zircon leach liquor using bis(2-ethylhexyl) phosphate, while Ma et al. (2020) examined basic sulfate precipitation from sulfuric-acid leach solutions. Botelho Junior et al. (2021) showed that Zr(IV) can be separated from Sc(III) in bauxite-residue liquors using organophosphorus extractants and TBP-containing mixtures, and Zhong et al. (2023) explored TRPO-modified adsorbents for selective Zr/Sc separation. These studies emphasize that "zirconium recovery" frequently means selective purification from another valuable metal rather than simple dissolution.

Impurity precipitation can also be process-enabling. Balinski et al. (2020) demonstrated in-situ precipitation concepts during eudialyte processing, and Pereira (2026) reviewed jarosite formation as an iron-control route in hydrometallurgy. These are transferable rather than zirconium-specific data, but they illustrate a design rule directly relevant to Zr liquors: inexpensive bulk-impurity removal should precede high-cost molecular Zr/Hf separation whenever the chemistry allows it.

6.2 TBP/nitric systems as the benchmark

The nitric-acid/TBP system remains the clearest benchmark for nuclear-grade Zr/Hf separation, consistent with established solvent-extraction and Zr/Hf separation principles (Rydberg, 2004; Banda & Lee, 2015). Amaral and Morais (2020) showed that acidity strongly influences Zr and Hf distribution in TBP extraction and developed equilibrium relationships for nitrate complexation and TBP solvation. Amaral et al. (2020) applied TBP extraction to nitric liquor derived from alkali-treated zircon concentrate and demonstrated a route toward nuclear zirconium oxide. Ravi Kanth et al. (2020) complemented this process evidence with a thermodynamic model for reactive extraction of macro-amounts of Zr and Hf with TBP, while Rozana et al. (2025) revisited TBP/kerosene for zirconium oxychloride-derived products.

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Sreenivasulu et al. (2024) examined trialkyl-phosphate solvents using experimental, theoretical, and EXAFS approaches, underscoring that classical extractants still offer room for mechanistic optimization.

6.3 Selective extractants: moving from bulk extraction to molecular recognition

Recent solvent-extraction studies increasingly target preferential Hf extraction or higher Zr/Hf discrimination. He et al. (2021) reported a Hf/Zr separation factor up to 15.7 in the Cyanex 572–HNO_3 system, with Hf extraction dominated by cation exchange. S. Ma et al. (2021) investigated an amic-acid extractant in chloride solution; J. Wang et al. (2020) compared extractant performance; J. Wang et al. (2023a) and J. Wang et al. (2023b) developed dialkylphosphinic-acid systems for Hf-over-Zr extraction in sulfate media; and Luo et al. (2024) reported a phosphate-ester extractant designed to preferentially recognize Hf. J. Zhao et al. (2020) contributed a diphosphonic-acid extractant for HCl media; Sahoo et al. (2022) examined a binary tri-n-octylamine/Cyanex923 system in sulfuric acid; and Tian et al. (2025) examined high-concentration Zr extraction from sulfate solutions with trioctylamine.

Thiocyanate and ketone/amine systems remain particularly active. Wu et al. (2022a) used Aliquat 336 in thiocyanate medium; Wu et al. (2022b) investigated synergistic TOPO/N1923 extraction; Xiong et al. (2024) examined the mechanism of the MIBK–HSCN system; H. Zhao et al. (2024) compared MIBK homologues; and S. Chen et al. (2026) introduced an MIBK–HSCN–glycine system for Hf-over-Zr separation. The importance of these papers lies less in a single highest separation factor than in the growing recognition that aqueous complex formation and organic-phase solvation must be co-designed.

6.4 Resins, chromatography, membranes, and porous adsorbents

Solid-phase and membrane separations offer a route to reduce organic-solvent inventory and facilitate continuous operation. Das et al. (2020) functionalized silica with phosphoric-acid-based ligands for Zr/Hf adsorption; Fan et al. (2022) developed DMHMP Levextrel resin for extraction chromatography; Qin et al. (2021) supported phosphorus-functionalized PAMAM dendrimers on mesoporous silica; and Hu et al. (2025) and J. Wang et al. (2025) reported phosphinic-acid-modified Merrifield resins. Ismail et al. (2025) used an anion-exchange resin to

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

separate Hf from zircon leach solution, while Poernomo et al. (2026) evaluated a Dowex-1X8 prototype for sulfate-anion feed.

A second wave uses materials engineered for selective coordination. Tang et al. (2023) developed an ion-imprinted membrane for Hf enrichment from zirconium oxychloride solutions. F. Zhao et al. (2024) used P507-modified magnetic porous carbon; L. Yang et al. (2025) coupled MIL-101-DGA with betaine hydrochloride; Shu et al. (2026) developed a phosphonate-functionalized COF-derived carbon aerogel; and Wan et al. (2026) reported selective Hf adsorption on SBA-15 silica. Xu et al. (2024) further demonstrated trialkyl-phosphine-oxide-modified resins for Sc/Zr systems, while X. L. Yang et al. (2025) reviewed extraction-resin structures and performance. These architectures are attractive for modular operation, but capacity, mass-transfer rate, regeneration stability, and fouling must be demonstrated over many cycles before they can displace solvent extraction at scale.

6.5 Ionic liquids, DESs, and liquid membranes

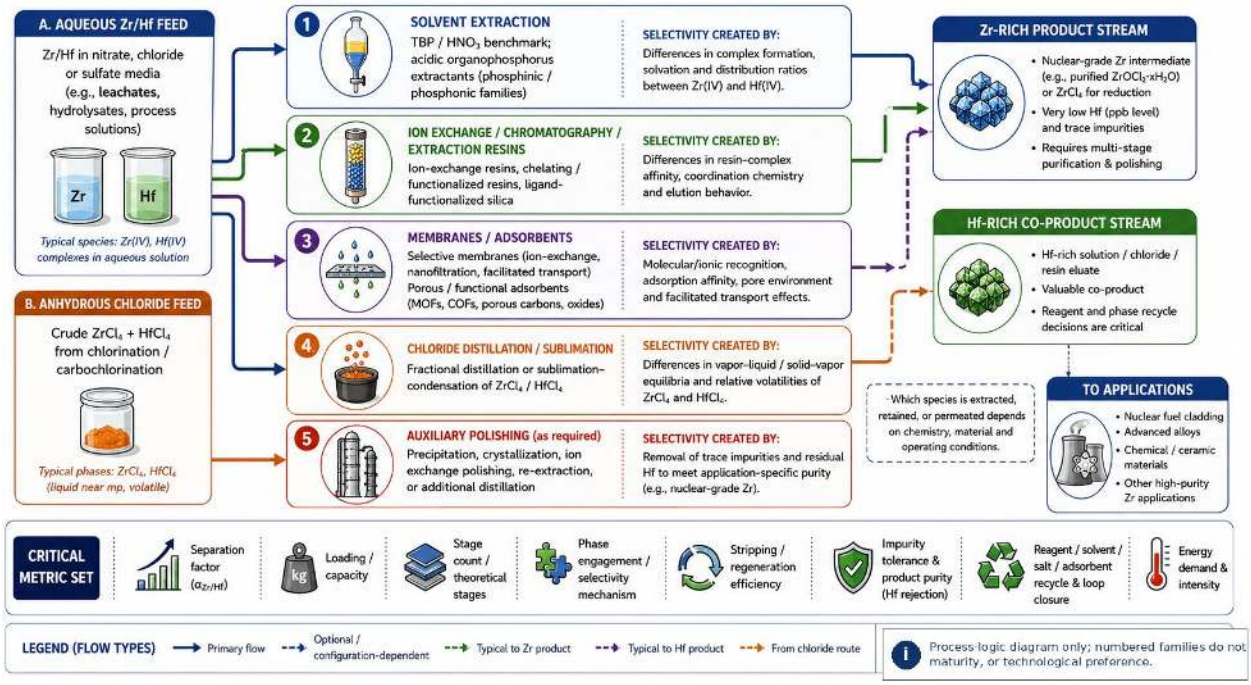
Task-specific ionic liquids and deep eutectic solvents (DESs) attempt to combine molecular selectivity with low volatility. Pathak et al. (2021) applied a task-specific ionic liquid to Zr/Hf extraction, and Tezcan and Donat (2022) used a multidropped liquid membrane with trioctylamine for selective Zr(IV) transport. Broader reviews of DES/ionic-liquid hydrometallurgy and critical-metal separation identify tunability and low vapor pressure as strengths but repeatedly flag viscosity, water sensitivity, extractant loss, toxicity uncertainty, and difficult solvent regeneration as scale-up constraints (Chen, Su, et al., 2025; Khalid & Santos, 2025; Schaeffer et al., 2025). Adjacent DES recycling studies reinforce the importance of temperature-dependent physical properties and closed-solvent operation when these media are proposed for zirconium systems (Chen, Shi, et al., 2025a; Shi et al., 2026).

Figure 3 positions the major Zr/Hf separation families according to where molecular selectivity is generated. This avoids the misleading comparison between a single-batch separation factor and an integrated purification train.

JIRRS
Journal International Review of Research Studies

Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Figure 3. *Zr/Hf separation options and the location of selectivity generation*



Note. Author-developed synthesis adapted from Amaral and Morais (2020), He et al. (2021), Tang et al. (2023), Luo et al. (2024), Ma et al. (2025), and Shu et al. (2026). Numbered families identify process logic only and do not imply ranking, maturity, or technological preference.

Figure 3 shows that all approaches ultimately trade selectivity against capacity, regeneration, and stage complexity. Solvent extraction offers high throughput and mature contacting equipment; solid adsorbents simplify phase separation but may have lower capacity or slower kinetics; membranes enable continuous enrichment but require chemical durability; and chloride distillation keeps the separation within a high-temperature chloride circuit. A credible flowsheet must therefore optimize the entire separation train rather than maximize a single equilibrium metric.

Table 3 condenses the recent separation literature into a process-oriented comparison.

JIRRS
Journal International Review of Research Studies

Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

1 **Table 3.** *Quantitative Cross-Comparison of Representative Zr/Hf Separation Technologies*

Technology/evidence	Representative feed or condition	Quantitative benchmark	Operability and scale-up interpretation	Representative evidence
TBP / HNO_3 — E3 direct	Nitric liquor; 2.5 mol L−1 TBP; A/O = 1:1 in reported tests	94% Zr extraction; αZr/Hf = 13.8 in one contact; continuous product ~99% Zr with ~200 mg kg−1 Hf	Continuous mixer-settler circuit reported. Mature contacting platform, but multistage acid/organic recycle, stripping, and entrainment remain process constraints.	Amaral & Morais (2020); Amaral et al. (2020); Ravi Kanth et al. (2020)
Cyanex 572 / HNO_3 — E2 direct	Zr 110 g L−1; Hf 3 g L−1; H^+ 2.6 M; nitrate 1.8 M; $(NH_4)_2SO_4$ 0.8 M	Hf extraction 76.8%; αHf/Zr up to 15.7	Batch equilibrium/mechanistic evidence. Promising selectivity; stripping and multicycle behavior remain to be validated for scale-up.	He et al. (2021)
DTBPPA phosphate ester — E2 direct	Acidic Zr/Hf system; ligand designed for preferential Hf recognition	Maximum reported αHf/Zr ≈ 16.3	Mechanistic evidence supported by spectroscopy/theory. Loading, extractant synthesis, and recycle determine process value.	Luo et al. (2024)
DMHMP Levextrel resin — E2–E3 direct	HNO_3–HCl mixed-acid extraction chromatography	αZr/Hf ≈ 47; recoveries 98–102%; mutual decontamination factor ~10^4	Column/extraction-chromatography demonstration. Strong selectivity signal; resin capacity and cycling remain decisive.	Fan et al. (2022)
Ion-imprinted membrane — E2 direct	Zirconium oxychloride solution containing Hf	Hf/Zr concentration ratio increased from 2.33:100 to 33.33:100 within 1 h	Short-duration membrane enrichment. Chemical durability and long-term flux/selectivity require validation.	Tang et al. (2023)
Porous adsorbents — E2 direct	SBA-15 and COF-derived carbon systems	SBA-15 βHf/Zr ≈ 2.9; COF-carbon αZr/Hf ≈ 3.3; capacities ~16.8 mg g−1 Zr and 12.0 mg g−1 Hf	Batch adsorption. Modular polishing potential, but capacity, kinetics, fouling, and regeneration govern scale-up.	Wan et al. (2026); Shu et al. (2026)

JIRRS
Journal International Review of Research Studies

Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Technology/evidence	Representative feed or condition	Quantitative benchmark	Operability and scale-up interpretation	Representative evidence
MIBK–HSCN–glycine — E2 direct	Thiocyanate/ketone system modified with glycine	Reported Hf/Zr separation coefficient ≈ 8.58	Batch extraction; illustrates co-design of aqueous complexation and organic solvation.	S. Chen et al. (2026)

Note. Author-developed synthesis from the cited literature. Separation factors are reported in the direction used by each study and are not directly interchangeable across media, feed compositions, phase ratios, and extraction directions. Numerical values are representative study conditions, not universal design values. The 98–102% recovery range reported for the DMHMP Levextrel resin is retained as a source-reported analytical/mass-balance result; values slightly above 100% should not be interpreted as a physical recovery greater than unity.

Table 3 reinforces a central conclusion of the hydrometallurgical literature: numerical selectivity must be interpreted together with operating mode and evidence maturity. The reported separation factors arise from different media, feed ratios, and selectivity directions and cannot be ranked as if they were measured under a common protocol. The most process-relevant evidence is therefore the combination of selectivity with loading or recovery, continuous or column operation, stripping/regeneration, impurity tolerance, and repeated cycles. The 2020–2026 shift toward engineered ligands and supports is scientifically significant, but industrial competitiveness will be decided by these coupled attributes rather than by the highest single equilibrium metric.

Table 4 reframes the Zr/Hf literature as a complete separation train. The purpose is not to reward the largest single-contact separation factor, but to show whether loading, scrubbing, stripping or elution, regeneration, impurity tolerance, repeated operation, and final product purity have all been demonstrated. Where the retained evidence does not support a process element, it is reported as not established rather than inferred.

Table 4. Process-Train Audit of Representative Zr/Hf Separation Technologies

Technology / evidence	Extraction or loading	Scrubbing	Stripping / elution	Regeneration / recycle	Impurity tolerance	Multicycle evidence	Demonstrated product purity
TBP/HNO_3 — E3 direct	94% Zr extraction; αZr/Hf = 13.8 in one contact under representative conditions.	Multistage purification is integral, but a standardized scrub performance is not consistently quantified across the retained papers.	Stripping is required; full strip efficiency is not consistently reported in the review evidence.	Acid/organic recycle is process-critical; long-term solvent-life and closed-loop losses remain incompletely documented.	Best supported for conditioned nitrate liquors; feed purification and entrainment control remain important.	Continuous mixer-settler circuit reported; full multicycle solvent-closure dataset is limited.	~99% Zr product with ~200 mg kg^{-1} Hf reported for a nuclear-zirconia intermediate.
Cyanex 572/HNO_3 — E2 direct	Hf extraction 76.8%; αHf/Zr up to 15.7.	Not established in the retained process evidence.	Not established in the retained process evidence.	Not established; multicycle extractant stability remains	Demonstrated in concentrated synthetic Zr/Hf nitrate media;	No integrated multicycle circuit demonstrated in	Selective Hf/Zr split demonstrated; qualified final

JIRRS
Journal International Review of Research Studies

Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Technology / evidence	Extraction or loading	Scrubbing	Stripping / elution	Regeneration / recycle	Impurity tolerance	Multicycle evidence	Demonstrated product purity
				a scale-up requirement.	broader impurity tolerance is unresolved.	the reviewed evidence.	product purity not reported.
DMHMP Levextrel resin — E2–E3 direct	Extraction-chromatography column; αZr/Hf ≈ 47; source-reported recoveries 98–102%.	Column separation provides staged discrimination; a dedicated scrub balance is not fully reported in the review synthesis.	Elution is inherent to the column operation; regenerant-loop closure is not quantified in the retained synthesis.	Resin capacity, regeneration, and cycling remain decisive uncertainties.	Demonstrated in HNO_3–HCl mixed-acid media; tolerance to broader feed impurities remains to be established.	Column evidence exists; long-duration multicycle resin-life data remain limited.	Mutual decontamination factor ~10^4 reported; final specification-grade product not demonstrated.
Ion-imprinted membrane — E2 direct	Hf/Zr concentration ratio increased from 2.33:100 to 33.33:100 within 1 h.	Not applicable as a conventional solvent-extraction scrub; feed conditioning remains necessary.	Permeate/retentate separation rather than chemical stripping; post-separation recovery not established.	Membrane regeneration and long-term chemical durability not established.	Demonstrated for zirconium oxychloride solution containing Hf; broader matrix tolerance unresolved.	Short-duration enrichment only; long-term flux/selectivity cycling not demonstrated.	Enrichment demonstrated; no qualified zirconium product reported.
Porous adsorbents — E2 direct	Representative βHf/Zr ≈ 2.9 or αZr/Hf ≈ 3.3; capacities ~16.8 mg g^{-1} Zr and ~12.0 mg g^{-1} Hf in cited systems.	Not generally established as a separate process stage.	Elution/stripping not sufficiently demonstrated for process closure in the retained evidence.	Regeneration, fouling resistance, and capacity retention are major unresolved requirements.	Mostly controlled laboratory solutions; realistic impurity competition remains a key gap.	Predominantly batch evidence; repeated-cycle robustness is not yet established.	Selective adsorption demonstrated; no final specification-grade product reported.
MIBK–HSCN–glycine — E2 direct	Reported Hf/Zr separation coefficient ≈ 8.58 in batch extraction.	Not established in the retained process evidence.	Not established in the retained process evidence.	Solvent regeneration and loss control require validation.	Demonstrated in a thiocyanate/ketone system; realistic impurity tolerance not established.	Batch evidence; no integrated multicycle circuit demonstrated.	Selective split demonstrated; final product specification not reported.
$ZrCl_4$/$HfCl_4$ molten-salt distillation — E2–E3 direct	Purified chloride feed; representative relative volatility ~1.18 in the studied melt/composition.	Not applicable in the aqueous-extraction sense; vapor/salt conditioning serves the analogous purification function.	Distillate/bottoms separation rather than stripping.	Closed chloride/salt management is essential; engineering-scale recycle remains a central requirement.	Requires well-controlled chloride purity, corrosion-resistant containment, and vapor handling.	Process design/stage analysis available; integrated long-duration recycle evidence remains limited.	Nuclear-purpose separation modeled/studied; final qualified metal or sponge purity not demonstrated by the selected study.

Note. Author-developed process-train audit from the evidence summarized in Section 6 and Table 3. "Not established" means the retained evidence base did not support a process-level claim; it does not prove that the operation is impossible or absent from all external literature. Values are representative study conditions and are not directly rankable across media.

7. Electrochemical and Molten-Salt Processing Routes

7.1 Why molten salts are attractive for zirconium

Aqueous electrowinning cannot readily produce zirconium metal because water reduction and zirconium oxide/hydroxide chemistry intervene long before Zr(IV) can be reduced to metal. Molten chlorides and fluorides remove the aqueous stability constraint and allow electrochemical control of zirconium valence and deposition; classical fused-salt thermodynamics and transport data remain foundational to interpreting these systems (Janz, 1967). S. Li et al. (2021a) reviewed

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

zirconium metal preparation by electrolysis, and Mirza et al. (2023) placed molten-salt processing within a broader nuclear electrochemical context. Zhou et al. (2026) confirm that nuclear-grade zirconium preparation and recovery in molten salts remains an active research frontier rather than a closed historical topic.

7.2 Chloride-melt redox behavior and cathode engineering

The Zr(IV) reduction pathway is frequently multistep and strongly dependent on salt composition and electrode material. S. Li et al. (2020) investigated Zr(IV) redox behavior and Zr/Hf separation in LiCl–KCl. Han et al. (2020) used Sn-coated W electrodes; Ding et al. (2021) studied extraction on Cu; Han et al. (2021) used Fe electrodes; and Murakami and Hayashi (2022) examined electrochemical recovery of Zr and Cd from molten chlorides for nitride-fuel reprocessing. Y. Wang et al. (2023) studied mixed Zr/Y chemistry in NaCl–KCl–K_2ZrF_6–YCl_3, while Zhang et al. (2022) investigated the mechanism of Zr(IV) electroreduction in NaCl–KCl.

Pulsed deposition offers a way to control nucleation and suppress unwanted lower-valence zirconium compounds. Cha and Yun (2023) demonstrated pulsed-potential electrodeposition of α-Zr from a practical $ZrCl_4$ concentration in LiCl–KCl, reporting current densities above 10^3 A m^{-2} while improving selectivity and adhesion. Mao et al. (2025) revisited fluoride regulation of zirconium-ion electrochemistry, and Mao et al. (2026) examined cathodic nucleation in NaCl–KCl. These studies collectively show that electrolyte anion chemistry and potential waveform are process variables, not merely electroanalytical details.

7.3 Consumable anodes and direct oxide-derived routes

Consumable-anode concepts attempt to integrate oxide conversion with metal deposition. S. Li et al. (2021b) prepared zirconium metal using a zirconium oxycarbonitride anode; S. Li et al. (2021c) used a zirconium oxycarbide anode; Shang et al. (2021) developed in-situ ZrC_xO_γ; and Su et al. (2021) synthesized zirconium oxycarbide by electroreduction of ZrO_2/C. S. Li et al. (2025) continued this direction with anodic dissolution of consumable ZrC_xO_γ in NaCl–KCl melts. Y. Chen et al. (2025b) coupled CO_2 capture with consumable-anode electrolysis, illustrating how the carbon/oxygen balance of the anode could be integrated with gas management rather than treated as a waste stream.

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Other approaches combine carbothermal preparation and electrochemical finishing. W. Song et al. (2025) prepared metallic Zr from ZrO_2 using carbothermal and molten-salt electrochemical reduction, while N. Li et al. (2021) produced Mg–Zr alloys by direct electro-deoxidation of MgO–ZrO_2 in $CaCl_2$–NaCl. Dumaire et al. (2024) and Y. Song et al. (2024) provide thermophysical and preparation data for ZrF_4-containing fluoride melts, and Chamberlain et al. (2025) demonstrate zirconium electrodeposition on uranium nitride in a fluoride salt, thereby extending the relevance to nuclear reprocessing.

7.4 Electrorefining, pool behavior, and process integration

Scale-up requires understanding the complete electrorefiner, not only the zirconium half-reaction. Sohn et al. (2021) used computational modeling to design molten-salt electrorefining to recover high-purity zirconium from spent nuclear fuel. Fredrickson et al. (2025) analyzed cadmium-pool behavior in the INL Mk-IV electrorefiner, providing transferable insight into liquid-metal inventory and electrorefiner operation. Even where the study target is not zirconium alone, such process-level evidence is essential because cathode phase behavior, salt composition, and co-deposited metals determine whether zirconium can be recovered as a separable product.

7.5 Product harvesting and qualification in electrochemical routes

Electrochemical feasibility cannot be established from voltammetry, current density, or zirconium recovery alone. A deposit must be harvested as a separable and qualifiable product. Future studies should therefore report deposit morphology and adherence, the mechanical or chemical method used to detach the deposit from inert or reactive cathodes, residual-salt inventory after washing or vacuum treatment, co-deposited metals and lower-valence zirconium phases, and the resulting oxygen, nitrogen, carbon, and hydrogen contents where the target is metallic zirconium. Reactive cathodes additionally require an explicit dealloying or refining balance, while consumable-anode routes require carbon/oxygen transfer and anode-gas accounting (Han et al., 2020, 2021; S. Li et al., 2021b, 2025; Cha & Yun, 2023).

Product qualification must also be linked to the next manufacturing step. A deposit intended for sponge replacement or direct melting should demonstrate compatibility with consolidation, remelting, alloying, and downstream fabrication rather than merely metallic phase

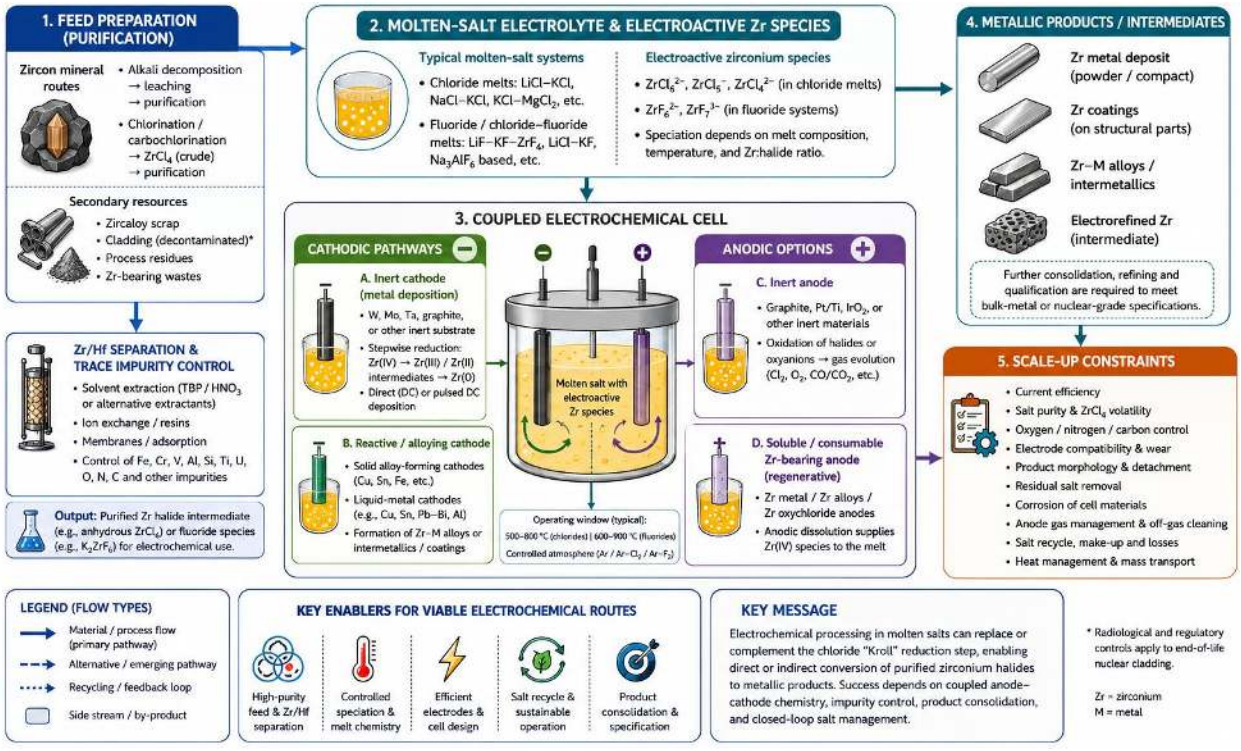
JIRRS
Journal International Review of Research Studies

Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

identification. For nuclear applications, the required evidence includes Hf control inherited from upstream separation, interstitials, residual halogens or salt, metallic contaminants, and analytical traceability against the applicable product specification. Coating studies provide useful transferable evidence on morphology and adhesion, but a coherent metal-production flowsheet additionally requires product recovery yield and a closed salt-cleaning loop (Y. Li et al., 2021, 2022; Chamberlain et al., 2025).

Figure 4 summarizes the electrochemical pathways and their scale-up constraints. The key distinction lies among deposition from soluble halide feeds, reactive cathodes that temporarily alloy with zirconium, and consumable anodes that convert oxide-derived intermediates within the electrolyzer.

Figure 4. *Electrochemical pathways for zirconium recovery in molten salts*



Note. Author-developed synthesis adapted from S. Li et al. (2020, 2021b), Han et al. (2020, 2021), Cha and Yun (2023), Y. Chen et al. (2025b), and Mao et al. (2025, 2026).

Figure 4 shows electrochemical routes are promising but not ready for full chloride–Kroll chain replacement. Their apparent simplicity is offset by salt purification, side reactions, product

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

separation, carbon/oxygen transfer from anodes, and the demonstration of product purity at relevant scales.

Table 5 compares representative electrochemical concepts using criteria relevant to industrialization.

JIRRS
Journal International Review of Research Studies

Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Table 5. Quantitative Operating Windows and Evidence Maturity for Representative Molten-Salt Routes

Concept/evidence	Representative operating window	Quantitative outcome	Product mode and process opportunity	Primary scale-up bottleneck
Pulsed inert-W cathode — E2 direct; Cha & Yun (2023)	LiCl–KCl with ~2.6 wt% $ZrCl_4$; pulsed-potential deposition	Current density >10^3 A m−2 with selective, adherent α-Zr reported	Metallic α-Zr deposit; waveform can suppress lower-valence products and improve adhesion	Salt purity, lower-valence Zr species, current efficiency, and deposit harvesting
Liquid Sn / Sn-coated W — E2 direct; Han et al. (2020)	LiCl–KCl–K_2ZrF_6 at ~853 K; 0.1 A galvanostatic or −0.85 V potentiostatic examples	Zr collected through Sn-assisted deposition; Zr/Sn2Zr phases reported	Reactive cathode shifts deposition thermodynamics and collects Zr as alloy/intermetallic	Downstream dealloying/refining and cathode inventory management
Fe reactive cathode — E2 direct; Han et al. (2021)	Molten LiCl–KCl; potentiostatic and galvanostatic recovery	Reported Zr recovery efficiencies 98.05% and 98.97%	ZrFe2-containing product; strong recovery signal from reactive cathode	Need for downstream alloy separation/refining
Oxycarbonitride anode — E2 direct; S. Li et al. (2021b)	Anode prepared near 1600 °C; NaCl–KCl electrolysis at about −2.9 V	Metallic Zr produced by coupled anodic dissolution/cathodic deposition	Integrates feed conversion with cathodic metal production	Anode-preparation energy, C/N/O transfer, gas handling, and current efficiency
ZrCxOy consumable anode — E2 direct; S. Li et al. (2025)	NaCl–KCl; studied cell-voltage window about 2.7–3.1 V	Metallic Zr obtained; fluoride and initial Zr concentration regulate behavior	Couples anodic dissolution with metal deposition	Integrated salt recycle, current efficiency, anode life, and gas treatment
Electrorefining flowsheet — E1 direct Zr / E4 transferable unit evidence	Molten chloride with liquid-metal pools; multicomponent nuclear context	Process-level modeling plus transferable Mk-IV electrorefiner operating evidence	High-purity recovery concept integrating salt and liquid-metal inventories	Multicomponent salt/pool control, co-deposition, product separation, and inventory management

Note. Author-developed synthesis based on direct zirconium evidence and explicitly identified transferable electrorefining evidence. Operating windows are representative of the cited experiments and should not be read as optimized industrial design values.

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Table 5 suggests that electrochemical routes should currently be judged as integration platforms rather than as isolated cell reactions. The most credible future systems will combine feed conversion, salt purification, controlled deposition, product harvesting, and anode-gas treatment in a closed circuit.

8. Secondary Zirconium Resources and Recycling

8.1 Scrap remelting and purification

Manufacturing scrap is the most straightforward secondary resource because it has already passed primary mineral conversion and, in many cases, Zr/Hf purification. Pereira et al. (2020) demonstrated the technical viability of vacuum induction and electron-beam melting for zirconium alloy scrap, while Vutova et al. (2025) examined the purification effect of electron-beam melting on recycled zirconium. Sohn (2025) reviewed smelting and recycling of zirconium more broadly. The key metallurgical issue is that remelting conserves alloy value only if interstitial oxygen, nitrogen, hydrogen, and volatile/nonvolatile alloying elements can be controlled; otherwise, the recycled metal may be downgraded even when overall zirconium yield is high.

8.2 Nuclear cladding recycling

Used-fuel cladding is more complex because zirconium recovery must be integrated with radiological decontamination and fuel-cycle safety. Hydrochlorination (Borjas Nevarez et al., 2021), low-temperature sulfur-chloride chemistry (Vestal et al., 2023; Conrad et al., 2023), electrorefining (Sohn et al., 2021; Murakami & Hayashi, 2022), and direct extraction concepts reviewed by Mondal and Mayes (2026) all seek to exploit the large mass fraction of zirconium in cladding while reducing high-level waste volume. Pucciarelli et al. (2024) provide a life-cycle perspective on circular-economy strategies in the nuclear industry, reinforcing that decontamination and waste reclassification can dominate benefits even when the recovered material value is modest.

Regulatory boundary. This review does not establish universal clearance, reuse, release, safeguards, or waste-classification criteria for irradiated or potentially contaminated zirconium streams. Feasibility of such routes is conditional on jurisdiction-specific radiological

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

characterization, decontamination criteria, nuclear-material control and safeguards, waste reclassification, worker/public protection requirements, and regulatory acceptance of the intended product or disposal pathway (International Atomic Energy Agency, 2014). Consequently, a technically high zirconium yield cannot by itself establish that recovered cladding is eligible for unrestricted reuse or nuclear requalification.

8.3 Upcycling zirconium wastes to functional zirconates

Not every secondary zirconium stream needs to return to metal. Fu et al. (2025) converted zirconium-containing wastes to low-cost Na_2ZrO_3 for CO_2 capture, and Gamba et al. (2026) synthesized Na_2ZrO_3 from Zircaloy waste for the same functional application. These studies widen the definition of recovery from “back to virgin zirconium” to “preserve zirconium functionality at the highest practical value.” The approach may tolerate impurity levels that would be unacceptable in nuclear-grade metal production, thereby reducing purification intensity.

This hierarchy matters for circularity. The technically highest-purity recycling route is not always the environmentally preferable route. If a contaminated or compositionally variable feed requires repeated halogenation, extraction, and high-temperature reduction to return to nuclear-grade metal, direct conversion to a functional oxide may yield better resource efficiency. For route screening, a practical value-retention hierarchy is direct reuse or alloy remelting, followed by refined-metal recovery, recovery of a chemical intermediate, and functional upcycling, provided that contamination, safety, and product specifications permit the higher-value option. This is a decision hierarchy rather than a universal LCA ranking: a lower step can be environmentally preferable when decontamination or purification burdens required by the nominally higher-value route are disproportionate.

9. Comparative Assessment: Maturity, Selectivity, and Process Integration

The FSSCQ Framework requires route-level comparison to separate scientific novelty from process maturity and to follow how selectivity, impurity burden, and product qualification propagate across interfaces. In this review, maturity is assessed using the E1–E5 rubric defined in Section 2 rather than an implicit technology-readiness score. Chlorination/Kroll and alkali

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

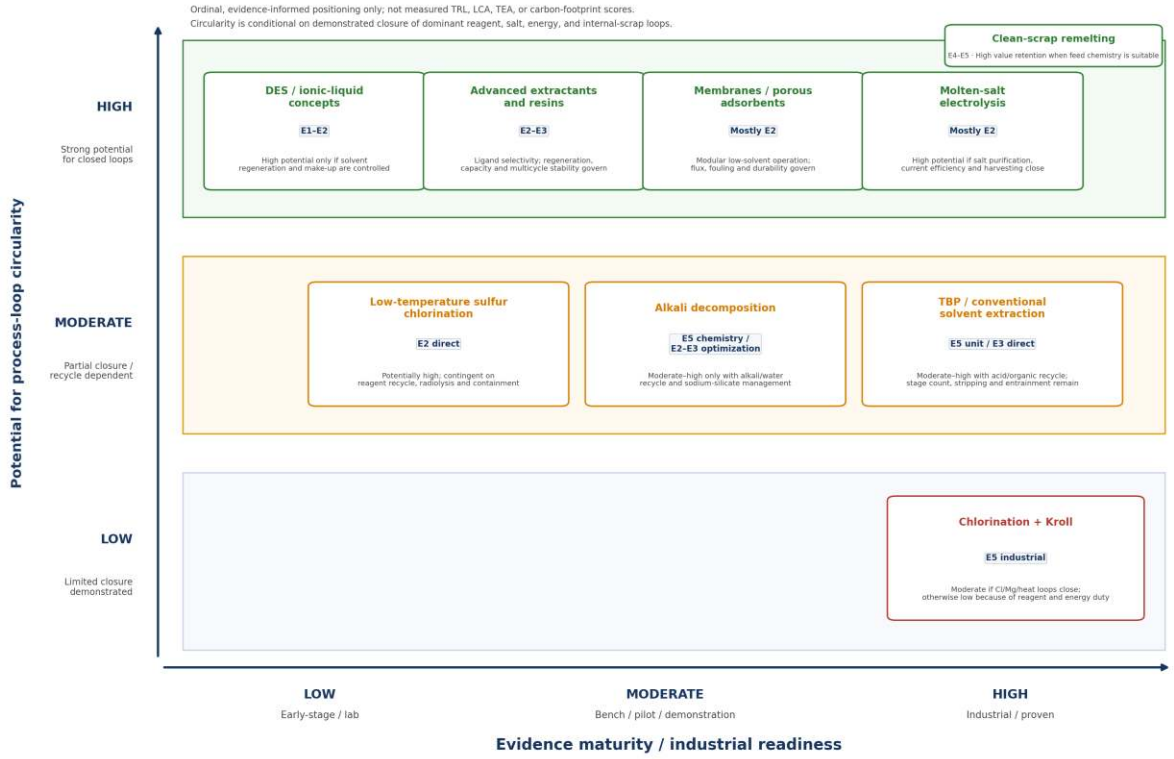
decomposition are anchored by established industrial practice, whereas recent solvent-extraction studies often combine mature contacting hardware with E2–E3 evidence for new ligands. Membranes, engineered porous sorbents, DES/ionic liquids, and most zirconium electrochemical concepts remain dominated by E1–E2 direct evidence, although some unit operations have higher transferable maturity. This separation between direct evidence and inherited unit-operation maturity prevents mature equipment from being mistaken for a mature zirconium-specific process.

Figure 5 places the main route families on an ordinal maturity–circularity map. The horizontal axis uses the evidence rubric in grouped form (low, moderate, high), while the vertical axis represents the potential to close major reagent, salt, or energy loops within a complete process boundary. The map deliberately avoids numerical 1–5 coordinates because the literature does not support pseudo-quantitative scoring, and it is neither an LCA result nor a formal TRL assessment.

JIRRS
Journal International Review of Research Studies

Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Figure 5. *Categorical maturity–circularity positioning of zirconium recovery routes*



Note. Author-developed ordinal classification using the E1–E5 evidence rubric and the process-level loop-closure criteria developed in this review. "High" circularity denotes potential contingent on demonstrated reagent, salt, and energy closure; categories are not measured LCA or TRL scores.

Figure 5 illustrates the central trade-off without assigning unsupported numerical precision. Conventional chlorination/Kroll has high evidence maturity but only low-to-moderate intrinsic loop-closure potential unless chloride, magnesium, heat, and by-product circuits are aggressively integrated. Clean-scrap remelting combines high maturity with high potential value retention when alloy chemistry is suitable. Advanced extractants and resins, membranes and porous adsorbents, DES/ionic-liquid concepts, and molten-salt electrolysis remain low-to-moderate in direct zirconium-specific maturity but can offer high circularity potential if solvent, sorbent, salt, and energy loops are demonstrated. Low-temperature sulfur chlorination remains E2 direct and is positioned conservatively at moderate present circularity because reagent recycle, radiolysis, and containment are not yet established at engineering scale. Apparent circularity is therefore conditional on salt purification, solvent or sorbent make-up, electrode replacement, emissions control, reagent recycle, and product requalification.

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Table 6 provides a route-selection matrix that can be used at concept-study level. The rankings are qualitative and should be replaced by site-specific mass and energy balances once feed assays and product specifications are defined.

Table 6. Critical Route-Selection Matrix for Zirconium Recovery

Route family	**Evidence maturity**	**Principal product endpoint**	**Where selectivity is generated**	**Circularity potential and decisive uncertainty**
Alkali decomposition	E5 chemistry / recent optimization E2–E3	Zr chemical intermediate or zirconia precursor	Zr/Si phase conversion; Zr/Hf deferred	Moderate–high only with alkali/water recycle; caustic inventory, washing, and sodium-silicate handling dominate
Chlorination + Kroll	E5 industrial reference	Nuclear-grade sponge / metal feed	Volatile chloride purification plus prior Zr/Hf separation	Low–moderate unless Cl/Mg/heat loops are closed; corrosion, $MgCl_2$ handling, and product qualification are decisive
TBP / conventional solvent extraction	E5 unit operation; recent direct circuits E3	Nuclear-purity Zr intermediate	Repeated nitrate complexation/solvation stages	Moderate–high with acid/organic recycle; stage count, stripping, entrainment, and waste-acid management remain
Advanced extractants/resins	Mostly E2–E3 direct	High-purity Zr/Hf split or polishing stream	Ligand-specific molecular recognition	Potentially high, but synthesis cost, capacity, stripping/regeneration, fouling, and multicycle stability remain uncertain
Membranes / porous adsorbents	Mostly E2 direct	Polishing/enrichment stream	Imprinted cavities and functionalized surfaces	Potentially high for modular low-solvent operation; flux, durability, capacity, and regeneration must be proven
Molten-salt electrolysis	Mostly E2 direct; some E4 transferable unit evidence	Zr metal / alloy / coating	Electrochemical valence and cathode/anode discrimination	Potentially high if conversion steps are removed; salt purity, current efficiency, electrode life, harvesting, and gas treatment govern

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Route family	Evidence maturity	Principal product endpoint	Where selectivity is generated	Circularity potential and decisive uncertainty
Low-temperature sulfur chlorination	E2 direct	$ZrCl_4$ from cladding	Selective low-temperature chlorination/decladding	Potentially high for nuclear-cladding mass reduction, but current loop closure is only moderate until reagent recycle, radiolysis, containment, and radiological qualification are demonstrated
Clean-scrap remelting	E4–E5 for suitable clean scrap	Recycled alloy/metal	Feed segregation and volatilization/refining; no Zr/Hf separation	High value retention with low reagent demand; interstitials and alloying-element buildup determine whether direct recycling is possible

Note. Author-developed assessment using the E1–E5 rubric defined in Section 2. Evidence levels describe the maturity of zirconium-specific evidence, while circularity potential depends on the effective closure of the dominant reagent, salt, energy, and internal-scrap loops.

Table 6 indicates that hybrid flowsheets are more defensible than single-technology narratives because the maturity of individual functions differs. A realistic primary route may combine E5 alkali opening with bulk impurity removal, an E3 direct Zr/Hf extraction circuit implemented in mature mixer-settlers, and E2 resin or membrane polishing. A secondary chloride route may combine selective cladding conversion with chloride purification and an electrochemical reduction step. The critical innovation frontier is therefore the interface between unit operations, where impurity deportment, reagent recycle, and product specification must remain compatible.

Product endpoints must be explicitly separated because a route adequate for zirconia or a functional zirconate may be unacceptable for reactor-grade metal. Table 7 therefore links representative nuclear-product composition controls to route-selection implications. The values are specification benchmarks, not universal design targets, and procurement standards and purchaser requirements take precedence.

Table 7. Product-Quality Targets Controlling Zirconium Route Selection

Product endpoint	Hf control	Interstitial controls	Other process-controlling impurities	Route-selection implication

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

Product endpoint	Hf control	Interstitial controls	Other process-controlling impurities	Route-selection implication
Reactor-grade virgin Zr / sponge (R60001)	Low-Hf reactor grade; active ASTM B349/B349M identifies Hf removal as defining	O, N, H and C must be controlled through feed purification, inert/vacuum handling, and melting practice	Residual halogens plus Fe, Cr, Ni and other metallic impurities are controlled by the applicable purchase specification	Zr/Hf separation must precede final metal production; reduction yield alone cannot establish nuclear-grade feasibility
Wrought unalloyed nuclear Zr, R60001 (ASTM B352/B352M-17(2021))	Hf ≤ 0.010 wt% (100 ppm)	H ≤ 0.0025 wt% (25 ppm); N ≤ 0.0080 wt% (80 ppm); O 0.09–0.15 wt% when specified by purchase order	Fe ≤ 0.150 wt% (1500 ppm); Cr ≤ 0.020 wt% (200 ppm); additional trace maxima apply	Shows that downstream qualification couples Hf separation with interstitial and metallic-impurity control
Nuclear-purity zirconium oxide/chemical intermediate	Recent TBP circuit reported product ~99% Zr with ~200 mg kg−1 Hf	Not a final-metal interstitial specification	Residual acid/ligand and co-extracted impurities must be compatible with subsequent chloride or oxide conversion	Intermediate purity must provide sufficient margin for final reactor-grade specification after downstream conversion
Functional zirconates / ceramic products	Application-dependent; no universal nuclear Hf limit	Application-dependent	Feed impurities may be tolerated if functional performance and safety requirements are met	Can avoid disproportionate purification when return to nuclear-grade metal would destroy the circularity advantage

Note. ASTM B349/B349M-16(2021) is the active specification for reactor-grade zirconium sponge/virgin metal and ASTM B352/B352M-17(2021) governs nuclear sheet, strip, and plate. The numerical wrought-product limits shown are for UNS R60001. Product-form standards differ; purchaser requirements and the current edition of the applicable standard must be used for qualification. Amaral et al. (2020) provide the representative nuclear-zirconia intermediate value.

10. Environmental, Energy, Economic, and Circularity Perspectives

10.1 Upstream mineral footprint and NORM

Environmental performance begins upstream. Corporate sustainability data and life-cycle studies of heavy-mineral production show that zircon's footprint depends on ore grade, mining method, mineral separation, electricity mix, and co-product allocation (Perks et al., 2022; Laghaei et al., 2025, 2026). Dvorzhak et al. (2025) and Mrdakovic Popic et al. (2026) emphasize that naturally occurring radioactive materials (NORM) require explicit inventory and impact-

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

treatment approaches in mineral-processing LCA. This is particularly relevant for heavy-mineral sands because monazite and other radioactive minerals may occur in the same separation circuit as zircon.

10.2 Reagent loops, residues, and energy carriers

The dominant environmental burden can move from mine to chemical plant as feed grade increases. Alkali decomposition generates sodium-bearing liquors and wash streams; chlorination requires halogen management and corrosion-resistant equipment; solvent extraction consumes acid/base for conditioning and stripping; electrochemical routes shift burden toward electricity, salt purification, and electrode materials. For zirconium, “closed loop” should therefore be defined by the fraction of the principal chemical carrier—NaOH/Na salts, chloride, fluoride, nitrate, or organic extractant—that is regenerated and reused over repeated cycles, not by a one-pass zirconium recovery percentage.

The same principle applies to impurity residues. Generic hydrometallurgical precipitation studies show that phase selection affects both metal losses and residue filterability (Balinski et al., 2020; Pereira, 2026). In zirconium systems, silica gels, iron precipitates, mixed zirconium basic salts, spent resins, and halide-containing solids can become hidden disposal burdens if not included in the flowsheet. This is one reason that direct conversion of suitable wastes to Na_2ZrO_3 or other functional products may outperform full purification to metal (Fu et al., 2025; Gamba et al., 2026).

10.3 Techno-economics and circularity metrics

Process-based techno-economic modeling for critical-mineral recovery demonstrates the importance of linking recovery performance to throughput, reagent consumption, equipment scale, and product value rather than using extraction percentage as an economic proxy (Baritto & Kumar, 2025). For zirconium, the economic model must also include the price premium associated with qualified nuclear products, the possible value of hafnium as a co-product, analytical certification, corrosion-resistant construction, and the cost of recycling or disposing of chloride-, fluoride-, nitrate-, alkali-, or organic-bearing streams. Trace chlorine control remains

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

relevant even after bulk chemistry appears to be within specification (Balaji Rao, Bano, et al., 2025).

To make circularity claims auditable, zirconium studies should distinguish zirconium yield from process-carrier closure and functional value retention. At minimum, the following normalized indicators should be reported when the necessary mass and energy data are available: saleable zirconium yield, Y_Zr = m(Zr in saleable product) / m(Zr in feed); dominant-reagent closure, C_R = m(reagent recycled) / [m(reagent recycled) + m(fresh reagent)]; fresh-reagent intensity, I_R = m(fresh reagent) / m(Zr recovered); process-energy intensity, I_E = E(process) / m(Zr recovered); dry-residue intensity, I_W = m(dry residue) / m(Zr recovered); and functional circularity, F_C = m(recovered Zr entering an equivalent-value application) / m(recovered Zr). These indicators extend the three-level logic of yield, reagent-loop closure, and functional circularity supported by nuclear-industry LCA and zirconium material-flow studies (Pucciarelli et al., 2024; Liu et al., 2026). Where a paper does not provide the data required for a metric, the correct entry is 'not reported' rather than an inferred value.

Metric status box. The indicators used in this review have different validation status and should not be interpreted as interchangeable evidence of environmental superiority.

Box 1. Status of Product, Process, and Circularity Metrics

Metric class	Examples / status	How it should be interpreted
Established product specifications	ASTM product-form chemistry and impurity limits; purchaser requirements.	Normative qualification criteria. Current applicable standards and purchase specifications govern acceptance.
Conventional process metrics	Extraction/recovery, separation factor, loading/capacity, current density/current efficiency, energy and residue quantities.	Established engineering or experimental descriptors when measured within a defined system boundary; they do not alone establish sustainability.
Proposed circularity-reporting indicators	Y_Zr, C_R, I_R, I_E, I_W, and F_C as defined in Section 10.3.	Author-proposed normalized reporting framework for auditable comparison across studies; not a validated predictive model.
Metrics requiring external validation	Route-level environmental superiority, economic viability, and net circularity advantage.	Require site/feed-specific TEA and/or LCA with explicit system boundaries, uncertainty treatment, and product-quality equivalence.

Note. The circularity indicators are proposed reporting variables intended to improve transparency. They are not validated predictors of lower life-cycle impacts or superior economics and therefore require future TEA/LCA validation before route-level environmental ranking.

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

11. Research Gaps and Future Directions

11.1 Report selectivity and operability together

The first research gap is reporting discipline. New extractants and adsorbents should report not only distribution ratios or separation factors, but also loading capacity, phase ratio, kinetics, stripping efficiency, solvent or sorbent regeneration, impurity tolerance, and performance over repeated cycles. The diversity of recent resins, membranes, MOFs, COF-derived carbons, silica supports, and ionic-liquid systems shows that molecular design is progressing faster than process validation (Hu et al., 2025; L. Yang et al., 2025; X. Yang et al., 2025; F. Zhao et al., 2024; Shu et al., 2026; Wan et al., 2026).

11.2 Reduce halogen intensity without losing intermediate quality

A second gap is the tension between halogen reduction and high-purity process intermediates. Fluorination of zircon can produce useful silica and zirconium fluorides, as shown by Y. Wang et al. (2020), but fluoride chemistry increases corrosion and environmental complexity. Chloride routes produce a highly useful volatile intermediate but demand chloride recycle. Future work should therefore compare “halogen avoided” against “separation work displaced.” A process that uses less halogen but creates a dilute, impurity-rich aqueous stream may simply move the burden to solvent extraction.

11.3 Demonstrate integrated electrochemical flowsheets at meaningful scale

Electrochemical research should move from voltammetry and gram-scale deposits toward continuous or semi-continuous cells with measured current efficiency, impurity balances, salt recycle, electrode life, and product harvesting. The literature now contains enough mechanistic information on cathode selection, fluoride effects, nucleation, and consumable anodes to justify integrated demonstrations (Ding et al., 2021; S. Li et al., 2020, 2021b, 2021c, 2025; Cha & Yun, 2023; Mao et al., 2025, 2026). Coating studies also show that pulsed-current control can produce functional zirconium deposits, providing transferable knowledge on morphology and adhesion (Y. Li et al., 2021; Y. Li et al., 2022).

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

11.4 Treat secondary resources as feed-specific metallurgical products

Secondary zirconium research should be segmented by feed. Clean manufacturing scrap should prioritize direct remelting and alloy-property retention; nuclear cladding should prioritize selective decladding, decontamination, and waste-volume reduction; complex hydrometallurgical residues should prioritize bulk impurity removal before Zr/Hf purification; and low-grade zirconium wastes should be screened for direct conversion to zirconates or ceramics. This hierarchy is supported by the divergent evidence from scrap melting, hazardous uranium residues, cladding chlorination, and zirconate upcycling (Pereira et al., 2020; Fan et al., 2023; Vestal et al., 2023; Fu et al., 2025; Gamba et al., 2026).

11.5 Use common sustainability and evidence-maturity metrics

Finally, future papers should report system boundaries and evidence maturity explicitly. Heavy-mineral LCA, NORM inventory work, nuclear circularity studies, and zirconium material-flow analyses provide a foundation for common indicators, but extractive-metallurgy papers still rarely connect laboratory chemistry to life-cycle consequences (Laghaei et al., 2025, 2026; Dvorzhak et al., 2025; Mrdakovic Popic et al., 2026; Pucciarelli et al., 2024; Zhu et al., 2023). At minimum, new zirconium recovery studies should report reagent intensity per mass of recovered Zr, energy input, waste/residue generation, recycle ratio for the dominant process chemical, purity achieved, Zr/Hf separation performance, and the scale/evidence level of the experiment. Claims of nuclear-grade feasibility should additionally report the product form and applicable specification, especially the achieved Hf concentration and the interstitial/metallic impurities that can be altered by chloride, electrochemical, or remelting steps.

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

12. Limitations of the Review Method

This review is a structured critical narrative synthesis, not a PRISMA-compliant systematic review. The final 111-paper analytical corpus, eligibility logic, Boolean architecture, evidence directness, and source-level coding are auditable; however, original database hit counts, duplicate-removal records, and article-by-article exclusion logs were not preserved and cannot be reconstructed independently. Database coverage may therefore reflect the indexing and search-field biases of Scopus, Web of Science, and ScienceDirect, even though database-specific syntax was adapted and complementary search blocks were used.

Evidence-maturity coding is another explicit boundary condition. E1–E5 assignments were made by a single reviewer and no independent human second-coder reliability statistic was available; the classifications are therefore interpretive maturity judgments, not reliability-tested measurements. The rubric ranks integration and industrial evidence rather than intrinsic methodological quality. Some sources provide transferable non-zirconium-specific evidence for unit operations, sustainability, NORM treatment, or process modeling; such evidence is labelled transferable and is not used to claim zirconium-specific performance. Reporting-completeness fields in Supplementary Table S1 expose missing regeneration, multicycle, impurity-tolerance, and product-qualification information instead of imputing it.

The August 2026 cutoff improves currency but creates an additional indexing boundary for very recent publications. Early-online records were retained only when stable bibliographic metadata, DOI or permanent publisher traceability, and full text were available by the cutoff. Finally, the maturity–circularity map and the FSSCQ Framework are conceptual synthesis tools: they support conditional process benchmarking and identification of evidence gaps, but they are not statistical models, formal TRL scores, LCAs, or validated predictors of environmental or economic superiority.

13. Conclusions

Under the Feed–Speciation–Selectivity–Conversion–Qualification (FSSCQ) Framework, zirconium recovery is best understood as a sequence of coupled selectivity problems rather than

JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

as a single extraction operation. Refractory zircon chemistry determines the severity of the front end, but Zr/Hf separation and trace-impurity control frequently determine whether the product can enter high-value or nuclear applications. Conventional alkali and chloride routes remain strong because they generate concentrated, controllable intermediates; their future competitiveness depends on improved reagent-loop closure, energy integration, corrosion control, and residue management.

Hydrometallurgical research has shifted from conventional solvent extraction toward molecularly designed extractants, resins, membranes, and porous adsorbents. These technologies can increase selectivity, but the decisive industrial metrics are regeneration, capacity, kinetics, stability, and continuous operability. Molten-salt electrochemistry is the most strategically important route for process intensification because it can couple conversion and metal production, yet it still requires integrated demonstrations that include salt purification, current efficiency, electrode life, impurity deportment, and product recovery.

Secondary zirconium resources should be routed according to feed quality and end-use value. Clean scrap favors direct metallurgical recycling; nuclear cladding requires selective chemical or electrochemical decontamination; and heterogeneous wastes may be better upcycled into zirconates than purified back to nuclear-grade metal. The most credible future flowsheets will therefore be hybrid, combining mature front-end chemistry with targeted molecular separation or electrochemical finishing while closing the dominant reagent and salt loops.

The principal research priority is to replace one-pass performance claims with integrated evidence. Recovery, purity, Zr/Hf separation, reagent recycle, energy use, residue generation, product qualification, and technology maturity should be reported together. Claims of nuclear-grade feasibility should be tied explicitly to the product form and to quantitative hafnium, interstitial, metallic-impurity, and residual-halogen requirements rather than to zirconium recovery alone. Progress on these coupled metrics will determine whether emerging zirconium technologies become industrial alternatives or remain laboratory demonstrations.

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JIRRS
Journal International Review of Research Studies
Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
DOI: 10.66104/s3mdb246
Pages: 1-58

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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JIRRS
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JIRRS
Journal International Review of Research Studies

Received: 04/08/2026 - **Accepted:** 05/09/2026
Vol: 01.09
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Pages: 1-58

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