Reporting Practices, Evidence Transparency, and Reproducibility in Spent NiMo/NiMoV Catalyst Recovery: A Perspective on Auditable Process Claims

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ABSTRACT

Published work on metal recovery from spent NiMo/NiMoV hydroprocessing catalysts often reports dissolution or stage yields, whereas evaluating a qualified recovered product requires additional accounting boundaries, reconciled inventories, and product-quality evidence. This Perspective proposes a claim-specific framework for interpreting the evidential strength of such reports. An inherited collection of 106 bibliographic records informed topic selection and illustrative examples; it was not subjected to a reproducible systematic search or full-text coding, and its size must not be interpreted as the number of appraised studies. We distinguish feed characterization, pretreatment, reaction evidence, analytical quality control, elemental closure, uncertainty, and product qualification, and operationalize an applicability-aware A–D reporting rubric. A hypothetical worked example illustrates how the rubric would be applied without assigning empirical grades to publications. The contribution is a testable minimum reporting dataset, clear distinctions among extraction, recovery, selectivity and purity, and sequential evidence gates for laboratory and process-development claims. This article makes no estimate of disclosure frequencies or route-level performance superiority. A subsequent evidence-mapping review would be needed to test prevalence, inter-rater reliability, and discriminative validity across verified full texts.

Keywords: spent hydroprocessing catalysts; NiMoV recovery; reporting transparency; reproducibility; elemental mass balance; evidence quality

Highlights

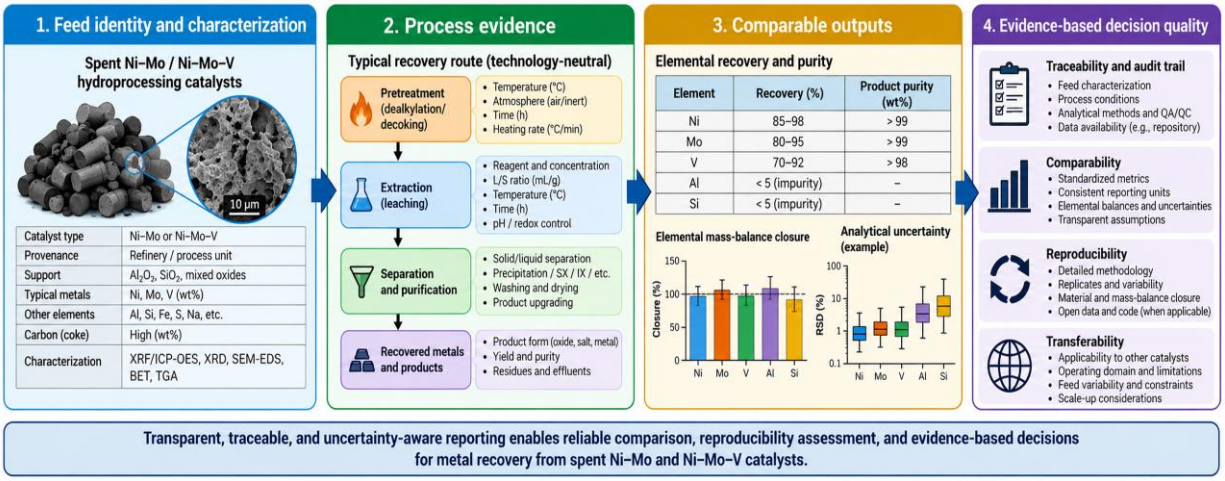
• Recovery claims require explicit system boundaries and element-specific denominators.

• Feed characterization and experimental conditions determine comparability.

• Mass closure, uncertainty, and product quality form a minimum evidence chain.

• A four-level reporting rubric enables transparent evidence screening.

Graphical abstract



INTRODUCTION

Spent hydroprocessing catalysts contain residual petroleum-derived organics, sulfides, support oxides, and potentially valuable metals. The value of a recovery percentage depends on the original feed, assay basis, accounting boundary, and qualified product. Processing-route reviews describe a wide range of technologies (Liang et al., 2022; Zhao et al., 2024; Gan et al., 2026), but their primary focus on technologies and recovery routes is distinct from the present contribution: a claim-specific framework for assessing whether reported evidence permits independent reconstruction of a performance claim. This Perspective is not a systematic review, scoping review, or quantitative meta-analysis.

The central question is not which isolated treatment reports the highest percentage, but what documentation permits an extraction, product-recovery, or scale-up claim to be independently assessed. Table 1 identifies evidence classes; Tables 2–4 propose disclosure requirements, definitions, and verification actions. Figure 1 outlines traceability, Figure 2 separates accounting boundaries, Figure 3 introduces the A–D appraisal rubric, and Figure 4 outlines evidence gates. The diagrams are conceptual and contain no observed study-level compliance data. The added operational decision rules and hypothetical example establish how the rubric could be tested in a future empirical review.

Specific experimental examples range from integrated leaching-and-separation studies to roasting and process optimization (Chen et al., 2022; Fang et al., 2024; Zhang et al., 2020). These publications motivate explicit distinctions among dissolved-metal extraction, recovered product mass, purity, and total process recovery. The objective is to establish a practical minimum reporting standard while retaining a clear boundary between bibliographic mapping and full-text evidence verification.

Perspective Development, Source Base, and Scope

Bibliographic source base and epistemic limits

The starting bibliography comprised 106 records supplied during manuscript development, with a small overlapping curated list. This count is a description of the inherited bibliography, not a PRISMA screening result, an independently retrieved study sample, or a full-text evidence-audit denominator. Individual records and associated metadata have not all been publisher-verified; publication-level eligibility, supporting-information availability, and full-text reporting completeness remain unassessed. No publication is assigned an empirical reporting grade in this article.

Sources from approximately 2020–2026 were used to develop examples and assess the relevance of potential reporting fields. Direct spent NiMo/NiMoV experiments, CoMo analogs, model-solution experiments, reviews, and techno-economic or environmental studies play different evidential roles and must not be pooled as equivalent test cases. Citations illustrate methodological issues but do not constitute a coded synthesis.

Because a search strategy, date-stamped database queries, independent eligibility decisions, and full-text matrix were not completed, no PRISMA flow diagram or incidence statistics are presented.

Development and intended future validation of the appraisal framework

The framework was constructed from the reporting chain required to reconstruct a process claim: a characterized original feed; measured pretreatment changes; documented experimental conditions; traceable analytical methods; reconciled elemental inputs and outputs; uncertainty; and, when claimed, a final qualified product. Each item must be coded as reported and verifiable, reported but insufficiently verifiable, not reported, or not applicable, with an exact page/table/supplement identifier recorded by future auditors. Coding must also preserve the claim type (dissolution, stage separation, integrated recovery, or pilot/industrial performance). A genuine future evidence map requires recorded searches, title/abstract and full-text screening, reasons for exclusion, independent coders, and agreement analysis.

The present Perspective contains no study-by-study extraction or compliance frequencies. Its rubric and gap matrices are prescriptive instruments. They are not empirical findings about the 106 cited records and should not be used to rank named studies until independently applied to the full text and supplementary data.

Evidence Classes and Scope of Published Claims

Studies should be stratified by (i) catalyst chemistry and physical state, (ii) recovery route, (iii) experimental scale, and (iv) outcome boundary. Laboratory leach tests, integrated product-recovery flowsheets, pilot plants, and life-cycle or techno-economic models represent different evidence types; merging them into one performance league table would discard essential qualifiers.

Table 1 distinguishes the scope of experimental observations from the evidence needed to support route-specific claims. This distinction is fundamental when comparing results from different process scales.

Table 1. Classification of published studies and evidence levels

Primary evidence	**Reporting risk**
Feed, reagents, kinetics, extraction	Limited downstream recovery evidence
Products, residues, recycles	Incomplete impurity and recycle tracking
Continuous operation, throughput, inventories	Confidentiality and variable feed
Assumptions, inventories, scenarios	Dependent on external experimental inputs
Literature synthesis	Not independent experimental confirmation

Note. Conceptual evidence classes, not verified grades assigned to individual studies.

A claim derived from a simulated sulfate solution is valid for the solution chemistry studied but cannot be silently promoted to a claim about heterogeneous spent NiMoV solids. Similarly, an industrial acid-production case informs operability without validating a distinct catalyst feed. This distinction is particularly important when identifying process readiness and selecting comparable controls.

Reviews and analogs offer methodological context, but their data cannot establish reporting compliance for a specific NiMo/NiMoV experiment without a claim-by-claim full-text audit (Pathak et al., 2022; Srour et al., 2021; Wang et al., 2024).

Novelty relative to process-route reviews

Existing reviews of spent-catalyst metallurgy focus primarily on process alternatives, resource recovery, and

purification technologies (Liang et al., 2022; Zhao et al., 2024; Gan et al., 2026). Those syntheses help organize process chemistry and available recovery options. The present contribution instead specifies the evidence needed to verify a particular claim at a declared boundary. This difference matters because a paper can establish excellent leach extraction without measuring final product recovery; similarly, detailed solution speciation does not establish original-feed representativeness or elemental mass closure. Novelty therefore lies in linking claim type to denominator, essential measurements, explicit non-applicability rules, and an auditable decision path—not in proposing a new recovery technology or claiming that earlier reviews uniformly omitted these issues.

Comparison criterion | Route-focused reviews: catalog flowsheets and performance endpoints | This Perspective: reconcile feed-to-product boundaries, require traceable elemental inventories, separate analytical completeness from process efficiency, and define conditional reporting grades. These distinctions describe differences in primary emphasis rather than verified deficiencies in particular earlier reviews.

Reporting of Catalyst Feed Characteristics

Composition and Provenance

Metal deportment and leaching response depend on parent catalyst formulation, service history, coke accumulation, sulfur content, support phase, and feed preparation. Minimum characterization should report original catalyst type, source and sampling protocol, lot mass, particle-size distribution, dry-mass basis, and individual concentrations of Ni, Mo, V, Co, Al, Fe, S, and C as applicable. Analytical methods and digestion completeness should accompany each concentration.

Analytical and Mineralogical Reproducibility

Distinguish bulk compositional data from mineralogical phase identification: X-ray fluorescence and inductively coupled plasma measurements quantify elements, whereas X-ray diffraction, microscopy, and local compositional mapping help interpret their occurrence. Discrepancies between total and phase-associated inventories should be explained rather than treated as measurement noise. Samples taken after thermal or solvent pretreatment cannot be compared against as-received assays without a recorded mass-loss correction.

The minimum source and operating metadata needed to interpret an experimental result are organized in Table 2. Fields should be applied according to the study objective and material characteristics.

Table 2. Minimum metadata for feedstock and experimental reporting

Mandatory fields	**Verification**
Lot, source, history, representativeness	Document sampling and split protocol
Metal assay, S, C, moisture, assay basis	Reference standards and duplicates
PSD, morphology, solid form	Reported method and size basis
Solvent, time, temperature, mass change	Residue and recovered oil inventories
T, t, pH/free acidity, S/L, oxidant	Measured-versus-setpoint distinction
Streams, washing, product grade	Full flow and assay records

Note. Proposed reporting checklist. PSD = particle-size distribution; S/L = solid-to-liquid ratio. Field applicability depends on route and feed.

Table 2 is deliberately a disclosure matrix, not a universal prescription for analytical instruments. The suitable

method depends on the process and analytical detection limits; the essential requirement is a transparent match between method and measurand. Figure 1 connects these inputs to confidence in the final performance claim.

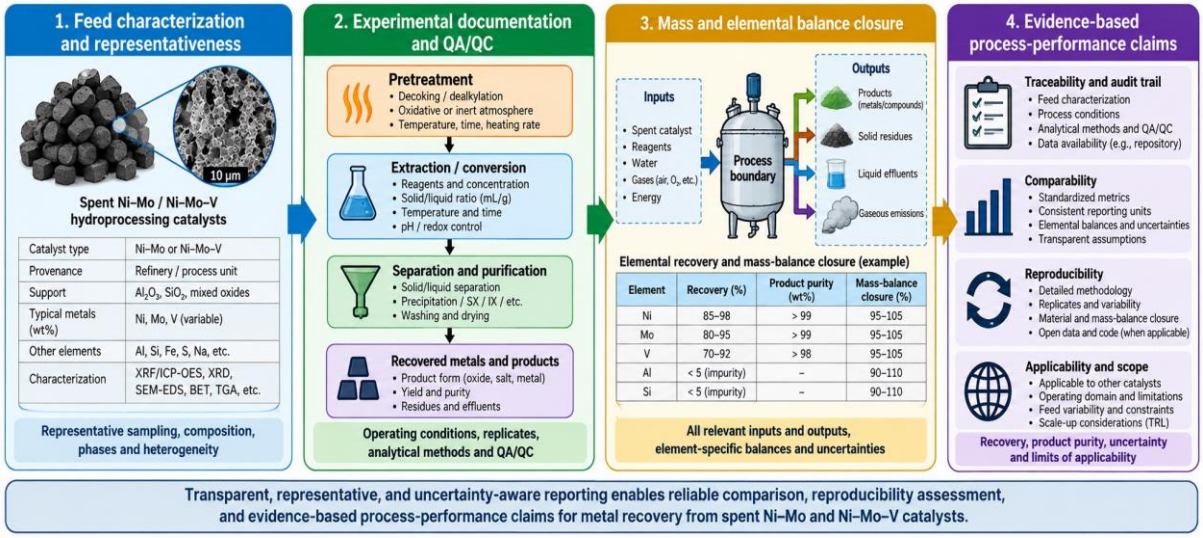


Figure 1. Traceable evidence chain for an auditable NiMo/NiMoV process claim.

Note. Original conceptual illustration; boxes denote required documentation, not measured results.

The diagram makes a methodological dependency explicit: accurate product assay does not compensate for an uncharacterized feed, and a well-specified reactor does not substitute for recovery-side material reconciliation. Each step requires independent documentation.

Reporting of Pretreatment and Operating Conditions

Oil removal, washing, thermal conditioning, and size reduction affect not only feed handling but also the basis for all subsequent metal recoveries. A pretreatment report should specify sample mass before and after treatment, solvent identity and dose, solid–liquid ratio, energy demand, wash number, gas atmosphere, and final residual carbon and sulfur. For oil-rich feeds, distinguish material recovered in the organic fraction from losses and volatilized matter.

Studies that specifically address petroleum fractions, solvent washing, and support conservation show why an upstream “cleaning” operation must be integrated into later mass accounting (Adavodi et al., 2025; Feng et al., 2022; Madej et al., 2025). When the impact of pretreatment is discussed, matched raw-versus-pretreated controls are preferable to comparing unrelated feeds.

For reactor operation, temperature should be defined as measured material temperature where feasible, not solely furnace setpoint; time zero and ramp schedule should be specified. The nominal reagent-to-solid ratio is incomplete unless reagent concentration, purity, and actual material moisture are available.

Reporting of Roasting and Leaching Results

Reporting Conversion and Reaction Exposure

Roasting studies need to report oxidation or reduction atmosphere, solids loading, residence-time distribution, additives, mixing, and gas handling. Leaching studies need actual liquid composition, ionic strength when important, solution-to-solid ratio, pH and/or free acidity, agitation, temperature, and assay sampling. A single optimized-condition endpoint cannot support a generalized mechanistic or kinetic model without time-resolved evidence.

Compare roasting and extraction routes as process families rather than ranking them directly by isolated single-

stage percentages; the breadth of oxidative, soda-roasting, and sequential treatment studies illustrates this (Arslanoğlu & Yaraş, 2021; Chen et al., 2024; Fang et al., 2024).

Kinetic and Mass-Transfer Claims

Fitted kinetic expressions must report the domain of temperature, particle size, mixing, solution composition, and fractional conversion used to obtain their parameters. A high coefficient of determination alone does not discriminate among chemical-reaction, pore-diffusion, and external-transfer controls. Residual diagnostics, independent validation, fit uncertainty, and identifiable model assumptions are therefore needed.

Reporting of Separation, Purification, and Product Quality

Metal extraction from a solid is not identical to recovery into a saleable oxide, salt, or intermediate. Separation papers should disclose raffinate, loaded organic/resin, strip solution, precipitate, mother liquor, washes, and purge streams. For each final product, grade must be based on an identified dry or wet mass basis, accompanied by impurity specification, product mass, and measurement method.

Multi-stage flowsheets—solvent extraction, ion exchange, staged precipitation, and conversion to oxides—are especially vulnerable to double counting if intermediate transfers are added as if independent recoveries (Chen et al., 2022; Han et al., 2023; Le & Lee, 2020). A product-grade claim also requires a defensible test method and representative composite sample.

Selectivity metrics should separate distribution selectivity in liquid-liquid systems, fractional precipitation, and product impurity rejection. A reagent may selectively dissolve Mo yet fail to produce high-purity MoO_3 after washing or calcination; both observations can be correct for different accounting boundaries.

Recovery, Selectivity, and Process Performance Metrics

Harmonized Denominators

For element i, extraction from feed into a designated solution should be defined as $E_i = (m_{i,solution} / m_{i,feed}) \times 100\%$. Overall elemental recovery should instead be $R_i = (m_{i,qualified\ product} / m_{i,original\ feed}) \times 100\%$, with any intermediate, recycle, and split streams explicitly accounted for. The definitions are not interchangeable. Where streams are recycled, the numerator must reflect net recovered product rather than gross internal circulation.

Table 3 establishes consistent definitions for the principal metrics reported in spent-catalyst recovery studies. These distinctions prevent single-stage dissolution yields from being interpreted as end-to-end recovery.

Table 3. Differences among recovery, extraction, selectivity, and purity measures

Recommended expression	Required boundary
$100 \times m_i$ in target liquor / m_i in leach feed	Single-stage dissolution
$100 \times m_i$ in qualified product / m_i in original feed	End-to-end process
100 × mass of target component/product mass	Defined material and assay basis
E_i / E_j, with conditions stated	Same feed, stage, and denominator
100 × total measured outputs / measured inputs	All output streams

Note. Definitions are proposed for harmonization; i and j denote elements, and masses require common dry/wet bases. Do not conflate distribution coefficients with extraction ratios.

As Table 3 summarizes, extraction at the leaching stage does not equal recovery of a qualified product from the original feed. A high dissolved fraction may coexist with material losses during separation, precipitation, washing, conversion, and purge disposal. Figure 2 explicitly separates the two denominators and places both

within an element-specific external material balance. Comparisons must remain qualitative when the published data do not allow these distinct quantities to be reconstructed.

Figure 2 displays the principal material pathways needed for a claim-specific recovery audit. Every box represents a reporting boundary rather than a particular industrial unit operation; routes that bypass pretreatment or leaching should adapt the same inventory logic.

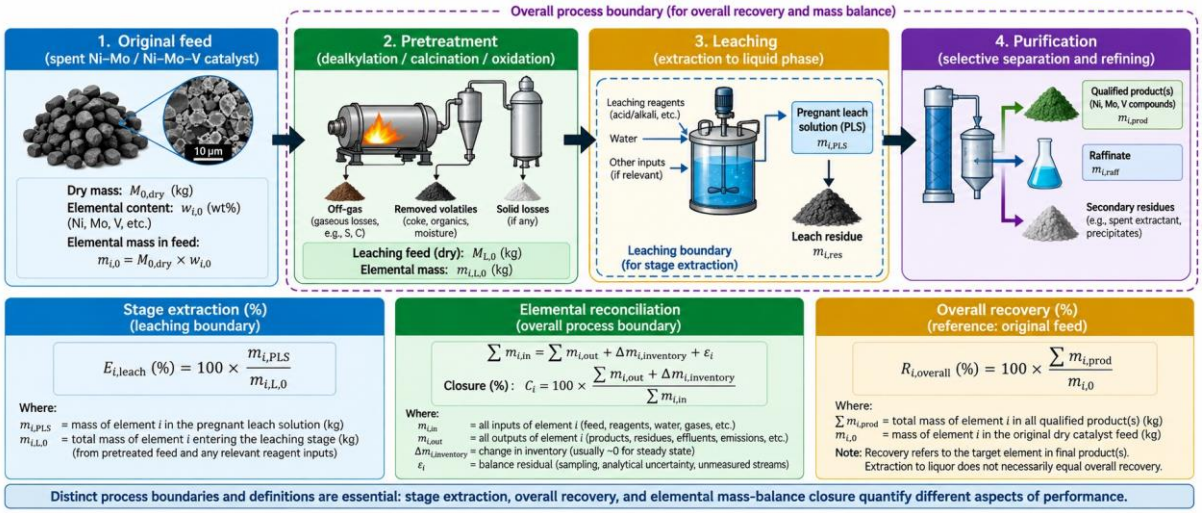


Figure 2. Elemental mass balance and accounting boundaries for Ni, Mo, and V extraction and recovery.

Note. Original conceptual mass-accounting schematic; no operating data or empirical recovery percentages are shown.

The essential implication is that an internal recycle is not a second recovered product. An auditable report distinguishes the elemental inventory entering each operation from net system outputs and provides explicit treatment of unsampled, retained, or unmeasured fractions.

Operating intensity and sustainability claims

Performance comparisons should also report reagent consumption per kilogram feed and per kilogram qualified product, water demand, heat and electricity demand, residue composition, and measured gaseous emissions. Claims of environmental superiority require a defined system boundary and inventory, rather than an isolated reduction in acid concentration or roasting temperature.

Reporting of Analytical Uncertainty and Mass Balances

A recoverability claim becomes auditable when every relevant element can be traced from feed into final products, residues, process liquids, and emissions. For an element i, the unreconciled residual is $\delta_i = m_{i,in} - \Sigma_k m_{i,out,k}$. Reporting δ_i alongside measurement uncertainty avoids replacing actual data with a forced 100% closure. Recycles should be represented consistently over the stated time basis and either excluded from the external balance or explicitly canceled.

Uncertainty originates in heterogeneous sampling, moisture corrections, assay calibration, incomplete digestion, liquid-volume determination, and residue retention. At minimum, authors should identify the replicate type (analytical, subsampling, or independently repeated process), sample size, central estimate, dispersion, and censoring of below-detection results. A precision figure without the replication hierarchy can be misleading.

A practical reporting framework should grade results by the completeness of the audit trail rather than the magnitude of recovery. Figure 3 shows four proposed levels; these are criteria for a future coded review, not empirically assigned frequencies.

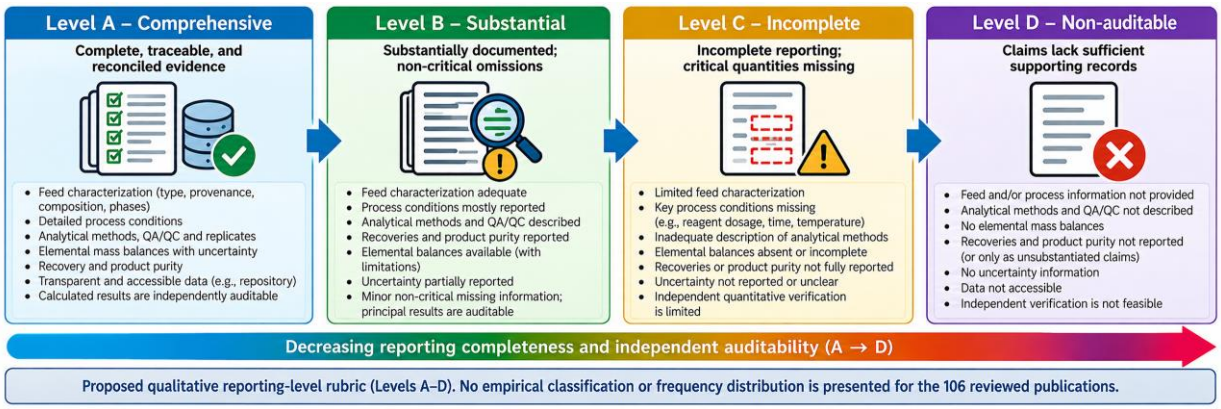


Figure 3. Four-tier reporting-quality rubric for NiMo/NiMoV recovery studies.

Note. Operational rubric for future appraisal; no publications in the bibliography have been graded.

The grading approach should not penalize a paper merely because an inapplicable analysis is omitted. Instead, mandatory fields must be adapted to its claim: a laboratory dissolution paper needs a defensible dissolved-metal balance, whereas a product-recovery or pilot-scale claim needs a wider process boundary.

Operational decision rules for the four-level rubric

Before grading, an assessor must record (1) the exact scientific claim, (2) the feed and stream boundary, (3) each applicable critical item, (4) its evidence location, and (5) whether evidence is independently reconstructable. Critical items for any quantitative leach claim are original feed assay and mass basis, leach conditions, calibrated metal assays, and a defined extraction denominator. Additional critical items for an end-to-end qualified-product claim are intermediate stream losses, net product mass and assay, product specification, and elemental reconciliation. Pilot-scale claims additionally require operating duration, throughput variability, and inventory boundaries. Items not applicable to a claim are excluded from the denominator, rather than coded as missing.

Proposed grading logic: Level A (auditable), all applicable critical items are reported and independently reconstructable, with uncertainty and relevant reconciliation; Level B (substantially documented), the principal outcome is reconstructable, and uncertainties are partly documented, but some non-critical supporting records are incomplete; Level C (partially documented), one or more critical inputs are missing, and the principal claim cannot be independently recalculated, although relevant measurements are reported; Level D (non-auditable), the claimed metric lacks an identifiable numerator, denominator, analytical basis, or underlying supporting records. A grade addresses reporting completeness for a specified claim, not chemical performance, scientific merit, or technology maturity. A missing method may cause C/D without implying that the actual process failed.

Hypothetical Worked Application (not a literature result)

Consider an invented laboratory report stating “95% Ni recovery” after leaching. It provides as-received feed mass and Ni assay, leach conditions, solution volume, Ni solution concentration, and replicate assays, but no downstream product isolation. The reported numerator and denominator could permit calculation of leach extraction; the 95% value must therefore be described as extraction rather than qualified-product recovery. Under the claim-specific rules, this record could receive Level A or B for the leaching-only claim if analytical uncertainty and stage balance are adequate, but Level C or D for an end-to-end recovery claim because no product mass, purity, or process reconciliation exists. This example is entirely hypothetical and does not grade any of the 106 bibliographic records.

A future pilot application should preregister item definitions and grade thresholds; have at least two reviewers code a purposive subset of eligible full texts independently; record disagreements, percent agreement, and a chance-adjusted coefficient where appropriate; refine the codebook; and then score the complete eligible set.

Item-specific reporting frequencies must be stated only after the audit, with denominators restricted to studies for which each item applies.

Critical Reporting Gaps and Evidence-Based Decision Framework

The stage-gated sequence in Figure 4 is intended as a decision aid for experimental reporting and independent assessment. Gates are conditional on the claim: a dissolution-only study need not document a saleable-product grade, but it must not label dissolution as end-to-end product recovery.

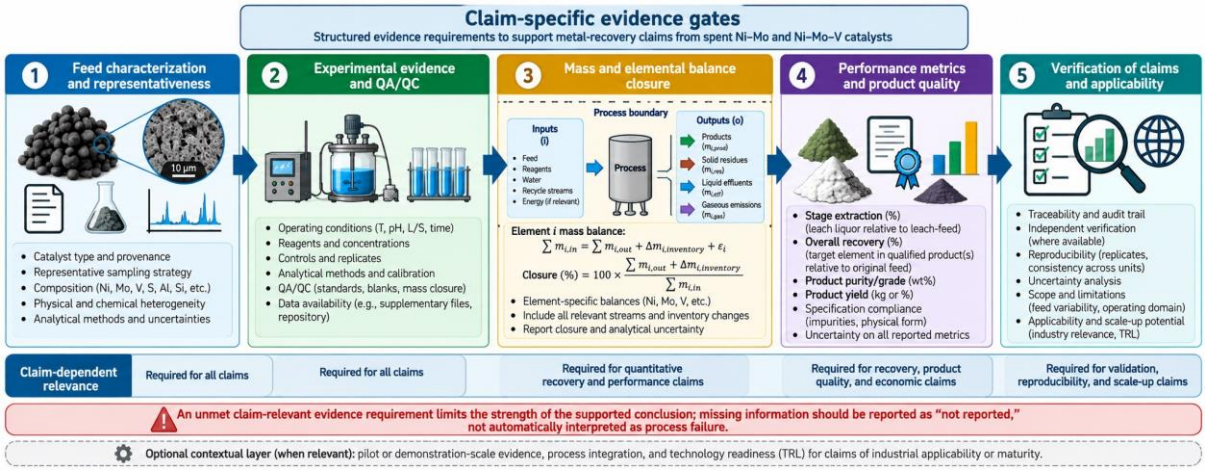


Figure 4. Stage-gated framework for claim-specific reporting quality and evidence validation.

Note. Proposed claim-specific evidence gates; no measured compliance values are presented.

Missing evidence should trigger a clearly qualified conclusion, not a presumption that the chemistry failed. Pilot and industrial claims can additionally require operating duration, throughput variability, environmental measurements, and independently reconciled inventories; technology readiness is informative only where those scale claims are supported.

Gap-to-Evidence Framework

A credible reporting standard is best structured as a compact minimum dataset, augmented by claim-specific data. Using it can reduce spurious comparisons, but this review cannot establish the prevalence of each omission without completing the planned full-text coding. The principal critical outcome is therefore a testable disclosure standard and a transparent limitation of the available evidence base.

Table 4 relates claim-specific reporting risks to the independent measurements required to resolve them. It is an assessment tool, not a quantified description of the reviewed corpus.

Table 4. Critical reporting gaps and recommended verification actions

Potential reporting gap	Minimum evidence action
Unknown lot-to-lot variation	Multiple batches and assay variance
Different assay bases	Mass-normalized paired tests
Missing gas, sulfur, and solids records	Full input/output inventory
Single fit without validation	Independent time series and model diagnostics
Purity without recovered mass	Product assays and elemental yield
Bench results used as industrial proof	Pilot continuity and measured balances
Incomplete life-cycle boundary	Transparent inventory and sensitivity

Note. Prospective verification actions, not documented defects or their frequencies among cited papers.

Table 4 organizes deficiencies as potential threats to interpretation, not allegations about particular studies. Techno-economic and environmental studies can be informative where their inputs and scenarios are transparent, but their assumptions should not be mistaken for independent experimental validation.

10.2 Technology maturity and evidence independence

Technology readiness level (TRL) terminology is useful selectively when a route makes pilot or deployment claims. Assigning a numeric TRL solely from a high bench extraction or from conceptual simulation would give a false impression of maturity. Process selection should instead reflect actual equipment scale, continuous-operation duration, feed variability, mass closure, product specifications, and independently verified economic assumptions.

The proposed disclosure rules create a testable basis for future independent assessment, but their predictive utility and inter-rater consistency have not yet been established empirically.

To convert this framework into a quantitative critical review, researchers should preregister a search protocol, retrieve verified full texts and supporting information, deduplicate by DOI, classify direct versus analog evidence, and independently code every field. Results should report item-level denominators, publication-year and route strata, missingness proportions with uncertainty, and sensitivity to exclusion of peripheral literature. The corpus should then be revised to retain only references that contribute materially to the evaluated reporting practices.

CONCLUSIONS

This Perspective proposes a claim-specific framework for making spent NiMo/NiMoV recovery reports auditable. Feed provenance, mass bases, pretreatment, experimental conditions, traceable assays, elemental reconciliation, uncertainty, and product-quality evidence are necessary in different combinations depending on the scientific claim. Extraction, separation yield, qualified-product purity, and end-to-end recovery are not interchangeable. The A–D rubric and its decision rules are proposed for independent testing; no study-level compliance statistics, systematic screening results, or comparative route rankings are asserted here. The next step is a reproducible, full-text evidence-mapping study with dual coding, explicit denominators, and rubric agreement testing.

Declarations

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No specific external funding was reported for preparation of this manuscript.

Conflicts of Interest

Conflict-of-interest disclosure must be confirmed by the author before submission.

Data Availability

No new experimental dataset was generated. The reference corpus was taken from the documents supplied for manuscript preparation; a paper-level evidence extraction matrix has not yet been completed.

Author Contributions

Conceptualization, literature synthesis, proposed reporting framework, visualization, and manuscript drafting: Antonio Clareti Pereira. The author should verify this statement prior to submission.

Use of Generative AI

Generative AI tools assisted language revision and conceptual illustration preparation. The author is

responsible for verification of all technical and bibliographic content and for meeting the target journal's disclosure requirements.

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