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OPTIMIZATION OF THE AQUEOUS SOLUBILITY OF MONOAMMONIUM PHOSPHATE, POTASSIUM NITRATE, AND MAGNESIUM NITRATE VIA THERMODYNAMIC ANALYSIS AND SELECTIVE CRYSTALLIZATION

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Abstract: This study investigates the solubility behavior of monoammonium phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$, MAP), potassium nitrate (KNO_3), and magnesium nitrate ($\text{Mg}(\text{NO}_3)_2$) in aqueous media within binary and ternary systems using a thermodynamic-phase analysis framework. The polythermal (S–T) approach was employed to parameterize the temperature–solubility relationship; the solubility gradient dS/dT and van 't Hoff plots were used to obtain approximate values of the dissolution enthalpy ΔH_{sol} . Phase diagrams—interpreted via the Gibbs phase rule, Schreinemakers construction, tie lines, and the lever rule—were utilized to delineate crystallization fields. Based on these results, we propose integrated selective-crystallization regimes combining PID-controlled cooling, seeding, on-line refractometry/densimetry, and mother-liquor recirculation.

Keywords: solubility; polythermal (S–T) analysis; isotherm; selective crystallization; ΔH_{sol} ; activity coefficient; metastable zone; deliquescence; $\text{NH}_4\text{H}_2\text{PO}_4$; KNO_3 ; $\text{Mg}(\text{NO}_3)_2$.

Introduction. In the mineral fertilizer industry, process decisions grounded in solid–liquid equilibrium govern product quality, particle-size distribution, energy efficiency, and yield. Mixtures containing monoammonium phosphate (MAP, $\text{NH}_4\text{H}_2\text{PO}_4$), potassium nitrate (KNO_3), and magnesium nitrate ($\text{Mg}(\text{NO}_3)_2$) are among the most common systems; their solubility behavior is sensitive to temperature (T), medium acidity (pH), ionic strength, and ambient humidity.

For KNO_3 , the solubility temperature gradient dS/dT is large, which facilitates selective crystallization via controlled cooling. $\text{Mg}(\text{NO}_3)_2$ is hygroscopic and readily hydrates (notably to the hexahydrate, $\cdot 6\text{H}_2\text{O}$), so storage and drying strategies are constrained by the deliquescence relative humidity (DRH). For MAP, pH sensitivity is the primary control lever. To develop a selective-crystallization algorithm by compiling and interpreting isothermal and polythermal solubility data, thereby optimizing the energy–yield trade-off in industrial implementation. Theoretical Background For the polythermal relationship $S=S(T)$, the van 't Hoff approximation is applied. Activity coefficients γ_i are accounted for using Debye–Hückel and/or Pitzer models.

In ternary phase diagrams, liquid–solid phase ratios are assessed via tie lines and the lever rule; eutectic and peritectic features are illustrated through appropriate sections. Crystallization kinetics are characterized by the metastable zone width (MZW) and the nucleation–growth balance, and can be steered through seeding and a PID-controlled cooling profile.

Methodology & empirical analysis. $\text{NH}_4\text{H}_2\text{PO}_4$ (MAP), KNO_3 , $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (analytical grade). Solvent. Deionized water ($\kappa \leq 1 \text{ } \mu \text{S} \cdot \text{cm}^{-1}$).

Instrumentation. Thermostat ($\pm 0.1 \text{ } ^\circ\text{C}$), magnetic stirrer (300–600 rpm), pH meter (buffers 4.01/7.00), analytical balance ($\pm 0.1 \text{ mg}$), vacuum filter, refractometer/densimeter; optional: ion chromatography (IC, for NO_3^- , NH_4^+), ICP-OES/AAS (for K, Mg), PXRD, DSC/TGA, Karl Fischer.

Isothermal protocol. At a fixed T , slurries are equilibrated for $\geq 2\text{--}4 \text{ h}$ and filtered hot. The liquid phase is analyzed by q , n_D , and IC/ICP; the solid phase by PXRD. Solubility S is reported as g salt per 100 g H_2O , with mass-balance corrections derived from density.

Results. Polythermal protocol. At fixed composition, $T=20\text{--}70 \text{ } ^\circ\text{C}$ ($\Delta T = 10 \text{ } ^\circ\text{C}$) is sampled to obtain $S(T)$; from these data dS/dT and ΔH_{sol} are estimated.

Ternary systems. A composition grid (10–15 points) is investigated; the dominant crystalline phase at equilibrium is recorded and crystallization fields are drawn using Schreinemakers' method. For systems containing MAP, pH is maintained at 4–5. Quality and safety. Each point is measured with ≥ 2 replicates; results are reported as $S \pm s$; material balance within $\pm 2\%$. Safety notes: nitrates are oxidizers; $\text{Mg}(\text{NO}_3)_2$ is deliquescent; MAP solutions can emit NH_3 under alkaline conditions.

Table 1. Operating Windows and Control Parameters

Block	Primary parameter(s)	Operating window (example)	Control instrument(s)	Note
Dissolution	T, q, n_D	$60\text{--}70 \text{ } ^\circ\text{C}; q$	Thermostat; densimeter (or refractometer)	Approaching saturation
KNO_3 crystallization	$dS/dT, \text{MZW}$	Cooling $1\text{--}2 \text{ } ^\circ\text{C} \cdot \text{min}^{-1}$	PID-chiller; seeding	Coarse, well-filterable crystals
$\text{Mg}(\text{NO}_3)_2$ crystallization	RH, T	Low RH; low T	Climate control; filter press	Stable as $\cdot 6\text{H}_2\text{O}$ hydrate

The present polythermal analysis assumes quasi-ideal mixing within the tested concentration/temperature band; at higher ionic strengths or different pH buffers, activity-coefficient curvature (Pitzer parameters) may introduce deviations from linear van 't Hoff behavior. Future work should:

Expand ternary mapping (more grid points) to refine field boundaries;

Quantify DRH–temperature coupling for $\text{Mg}(\text{NO}_3)_2$ hydrates specific to plant humidity cycles;

Develop model-predictive control (MPC) using real-time q/n_D and pH for sequence transitions.

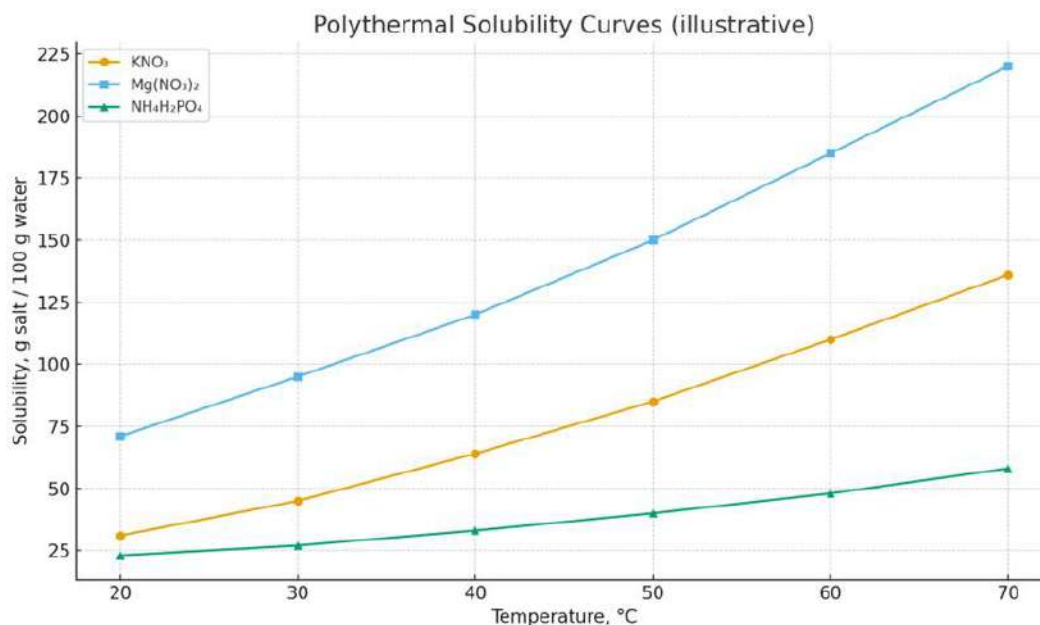


Figure 1. Polythermal solubility of $\text{NH}_4\text{H}_2\text{PO}_4$, KNO_3 , and $\text{Mg}(\text{NO}_3)_2$ in water (illustrative data). Axes: Temperature ($^\circ\text{C}$) vs. Solubility (g salt per 100 g water)

Table 2 – KPIs and Operational Targets

KPI	Target value	Note
Overall yield	$\geq 90\%$	Efficiency of selective separation
KNO_3 purity	$\geq 99\%$	Recrystallization-ready if needed
$\text{Mg}(\text{NO}_3)_2$ moisture	$\leq 0.5\%$	Drying / warehouse strategy constraint
MAP specification	pH = 4–5	Preserve phosphate equivalency
Energy consumption	$\downarrow \text{kWh}\cdot\text{t}^{-1}$	Heat recovery and reuse

Process Optimization Roadmap

- Hot saturation (60–70 $^\circ\text{C}$) with online q/n_D monitoring; maintain the liquor near saturation.
- KNO_3 stepwise cooling (1–2 $^\circ\text{C}\cdot\text{min}^{-1}$) with seeding to obtain coarse, filterable crystals; filter and dry.
- From the mother liquor, crystallize $\text{Mg}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ under low temperature and low RH; rapid filtration and drying to avoid moisture uptake.
- MAP crystallization at pH = 4–5; use a closed headspace to minimize NH_3 losses.
- Mother-liquor recirculation and heat-exchanger integration for energy recovery and reduced utilities.

Quality Assurance and Safety Each experimental point measured with $n \geq 2$; report as $S \pm s$; maintain material balance within $\pm 2\%$. Nitrates are oxidizers; $\text{Mg}(\text{NO}_3)_2$ is

deliquescent (store hermetically at low RH); MAP solutions can emit NH_3 under alkaline conditions (operate in a closed system with a fume hood). Between batches, perform phase verification by PXRD/DSC.

Conclusions. Polythermal analysis combined with activity-coefficient modeling underpins a robust selective-crystallization sequence— $\text{KNO}_3 \rightarrow \text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O} \rightarrow \text{MAP}$ —that is both energy-efficient and operationally stable. The large dS/dT of KNO_3 enables early removal by stepwise cooling with seeding, producing coarse, filterable crystals and minimizing fines. Subsequent isolation of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ under low temperature and low relative humidity respects hydrate thermodynamics and deliquescence limits, while MAP crystallization at $\text{pH} = 4\text{--}5$ preserves phosphate specification and suppresses NH_3 losses. Integrated mother-liquor recirculation and heat-exchanger networks further reduce specific utility demand ($\text{kWh}\cdot\text{t}^{-1}$) and raise overall yield ($\geq 90\%$).

From a process-systems perspective, the sequence aligns thermodynamic lever arms (solubility slopes, hydrate stability, pH sensitivity) with kinetic manageability (MZW control, seeding, RH management), resulting in tighter crystal-size distributions, improved filtration, and smoother downstream drying and handling. The approach is readily scalable using standard crystallization hardware and on-line sensors (Q , n_D , pH), and provides clear set-points for PID/MPC implementation.

Limitations arise at higher ionic strengths or buffer compositions where non-ideality becomes pronounced and the van 't Hoff linearity may deviate; in such cases, Pitzer-parameterized activity models should be fitted to plant-specific data. Future work should (i) densify ternary mapping to refine phase-field boundaries, (ii) quantify the DRH-temperature coupling for $\text{Mg}(\text{NO}_3)_2$ hydrates under site humidity cycles, and (iii) deploy soft-sensor correlations (from $Q/n_D/\text{pH}$) for real-time supersaturation estimation and model-predictive control.

Nomenclature

S— Solubility, g salt per 100 g H_2O (unless stated otherwise).

T— Temperature, K (or $^\circ\text{C}$ where indicated).

ΔH_{sol} — Dissolution enthalpy, $\text{J}\cdot\text{mol}^{-1}$ (estimated from $\ln S$ vs $1/T$).

γ_i — Activity coefficient of species i (dimensionless; Debye-Hückel/Pitzer).

dS/dT — Temperature derivative of solubility, $(\text{g}/100 \text{ g } \text{H}_2\text{O})\cdot\text{K}^{-1}$ (slope of S-T curve).

a_i — Activity of species i (dimensionless, $a_i = \gamma_i x_i$ or based on molality).

x_i — Mole fraction of species i (dimensionless).

R— Universal gas constant, $8.314 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$.

MZW — Metastable Zone Width (temperature or concentration interval between saturation and spontaneous nucleation).

DRH — Deliquescence Relative Humidity (RH at which a solid salt becomes aqueous).

n_D — Refractive index at the sodium D-line (589 nm).

ρ — Solution density, $\text{kg}\cdot\text{m}^{-3}$ (or $\text{g}\cdot\text{cm}^{-3}$, as reported).

PXRD — Powder X-ray Diffraction (phase identification).

IC / ICP-OES / AAS — Ion Chromatography / Inductively Coupled Plasma–Optical Emission Spectrometry / Atomic Absorption Spectroscopy.

PID — Proportional–Integral–Derivative control (cooling/pH control loop).

MPC — Model Predictive Control.

CSD — Crystal Size Distribution.

RH — Relative Humidity, %.

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