

# Reillex™ Resins

for metal recovery



A photograph of an industrial facility, likely a refinery or chemical plant, featuring several tall, cylindrical distillation columns and complex piping. The scene is set against a dramatic sunset sky with orange and yellow clouds. The lighting is soft, highlighting the metallic surfaces of the equipment.

# weak base resins

for metal recovery

Reillex Resins are crosslinked poly-4-vinyl-pyridine resins which are useful for adsorption and recovery of soluble metallic species from either aqueous or non-aqueous solutions. Reillex Resins differ from conventional alkylamine type weak-base resins because the thermally and chemically stable pyridine ring is one of the functional group present. Moreover, the unique structure of Reillex Resins results in other desirable properties such as a tough bead form, high porosity, and exceptionally high capacity.

**This brochure provides guidelines and general operating techniques applicable to metal recovery operations such as:**

- Treatment of electroplating wastewater
- Recovery of soluble catalysts from reaction mixtures
- Separation and purification of metal compounds
- Hydrometallurgical applications
- Precious metal recovery

In addition to solving metal recovery problems Reillex Resins have many other interesting uses.



# functionality

in solid form

Reillex 402 and Reillex 425 differ in physical form, level of crosslinking, and physical properties as noted in the tables. Of course, your choice between Reillex 402 and 425 resins depends on the particular application and process. Both Reillex Resins offer advantages which make them ideal for metal recovery applications.

## Stability

- Reillex Resins are stable up to 260°C for extended periods
- Conventional alkylamine weak-base resins have a maximum temperature of only 100°C
- The pyridine functional groups of Reillex are highly resistant to attack by oxidizing and reducing agents leading to longer resin life under harsh conditions
- A published study<sup>1</sup> indicates that vinylpyridine resins are more stable to ionizing radiation than other resin types. As a member of this family, Reillex Resins are expected to be excellent for these applications

## High Capacity

- The high pyridine ring content of Reillex Resins results in unusually high exchange capacity. High capacity means process advantages such as reduced equipment size, higher throughput, and reduced resin costs

## Versatility

- Reillex Resins adsorb a wide variety of metals from aqueous and non-aqueous solutions

## Other Features

- Odorless and noncontaminating to product and waste streams
- Now available in commercial quantity

Important Features of Reillex Resins	Reillex 402	Reillex 425
Tough physical form suited for batch reactions	•	•
Bead shape for column use	---	•
Porous, macroporous structure	•	•
Suitable for aqueous and nonaqueous applications	•	•
Insoluble in all solvents	•	•
High capacity	•	•
Stable at high temperatures	•	•
Broad pH stability	•	•
Low levels of soluble impurities	•	•

Typical Properties		Reillex 402	Reillex 425	
Appearance		off-white granular powder	off-white beads	
Particle size <sup>a</sup>		ca. 60 mesh	18-50 mesh	
Bulk density <sup>b</sup>	g/cm <sup>3</sup>	0.45	0.29	
	lb/ft <sup>3</sup>	28	18	
Skeletal density <sup>b</sup>	g/cm <sup>3</sup>	1.15	1.14	
Particle density <sup>b</sup>	g/cm <sup>3</sup>	---	0.6	
Surface area	m <sup>2</sup> /g	ca.0.5	ca.90	
Moisture retained upon filtration (% by weight)		36-39	50-60	
Approximate pK <sup>a</sup>		3-4	3-4	
Hydrogen ion capacity, in water (meq/g)		8.8	5.5	
% swelling from free base to hydrochloride form, in water		100%	52%	
	methanol	70-75	28-32	
	acetone	30-35	32-36	
	water	33-37	12-16	
	isopropanol	13-17	28-32	
	toluene	8-12	18-22	
	ethyl acetate	3-6	32-36	
% swelling from dry state to solvent - saturated state	hexane	0	12-16	
	Temperature Stability, maximum recommended for extended use	°C	225	260

<sup>a</sup> The particle size of Reillex 402 and 425 can be varied. Inquiries for material of different particle size will be considered

<sup>b</sup> Density determinations were carried out on dry material

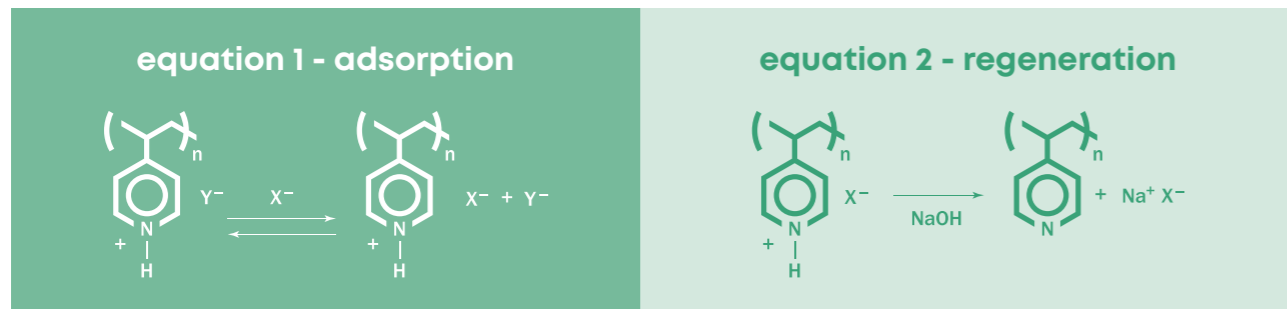
# extraction of metal ions

by Reillex Resins

Generally, each metal recovery problem is unique and requires individual treatment. The following guidelines should be helpful in applying Reillex Resins to your particular problem.

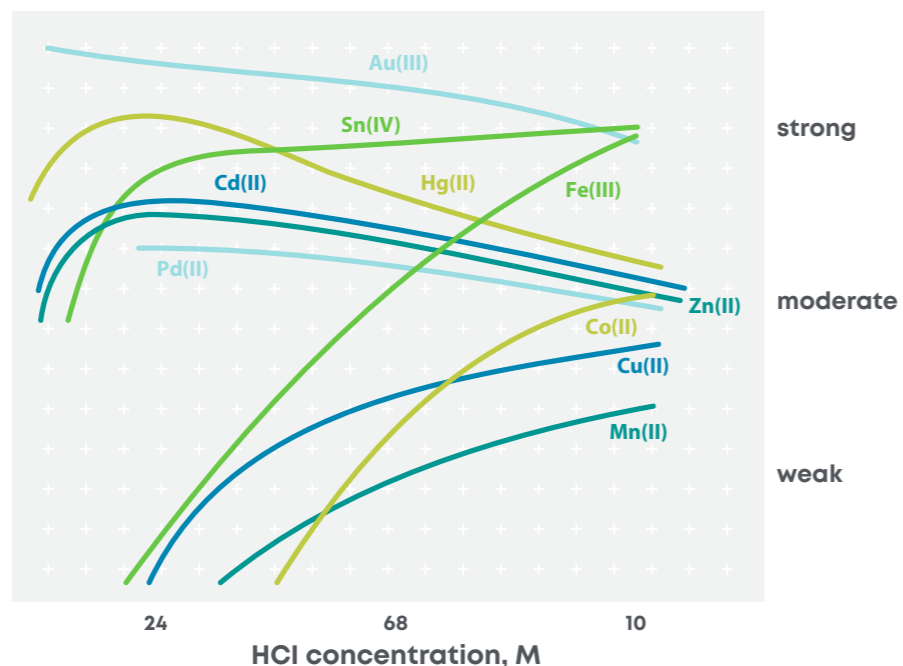
## extractions from acidic aqueous solutions

Reillex Resins function as weak base anion exchange resins in acidic solutions.<sup>2</sup> In the protonated form, Reillex Resins adsorb metals that are present as negatively charged complexes (eq.1). Regeneration is usually accomplished by neutralizing the anionic complex with base (eq.2).



## adsorption of metal ions by Reillex 425 resin from HCl solutions

The graph shows how the strength of adsorption of some metal ions varies with HCl concentration.<sup>3</sup>



## adsorption of metal ions by Reillex 425 resin in acidic systems

Ion Types	HCl	HNO <sub>3</sub>	H <sub>2</sub> SO <sub>4</sub>
V <sup>+5</sup>	strong	weak	strong
Cr <sup>+6</sup>	strong	strong	strong
Fe <sup>+3</sup>	strong	weak	moderate
Co <sup>+2</sup>	moderate	weak	weak
Ni <sup>+2</sup>	weak	weak	weak
Cu <sup>+2</sup>	moderate	weak	weak
Zn <sup>+2</sup>	strong	weak	weak
Ga <sup>+3</sup>	strong	weak	weak
Mo <sup>+6</sup>	strong	strong	strong
Pd <sup>+2</sup>	moderate	strong	weak
Cd <sup>+2</sup>	strong	weak	weak
Re <sup>+7</sup>	strong	strong	strong
Au <sup>+3</sup>	strong	strong	weak
U <sup>+6</sup>	strong	strong	strong

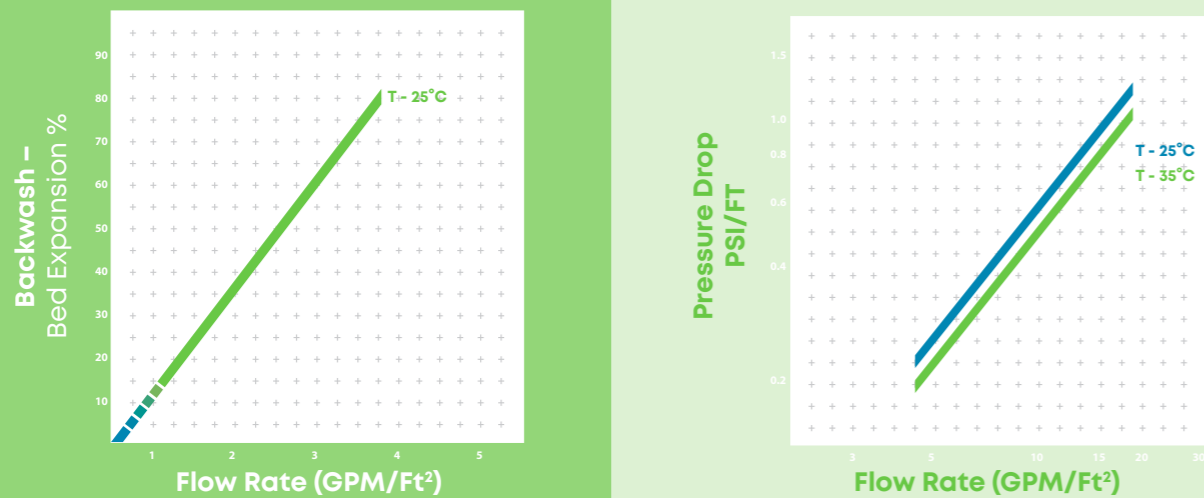
The table illustrates how metal adsorption depends on the nature of the acidic solution.<sup>4</sup> Anion exchange technology has been applied to many metal recovery problems. For more information about the anion exchange behavior of metals, see reference 4.



## extractions from neutral or nonaqueous solutions

Reillex Resins adsorb metal ions from neutral aqueous or nonaqueous solution through the formation of coordination complexes.<sup>5</sup> The strength of the complex depends on the particular metal ion involved, the solvent, and the other species present in solution. In some cases, metals can be separated based on differences in the strength of their coordination complexes.<sup>3</sup>

# general operating procedures



## Column or Batch Techniques

Metal recovery operations using Reillex Resins may be carried out using either column or batch techniques. The bead form of Reillex 425 is usually preferred for column applications, but the choice of resin and method depends on the specific application. Because it has exceptionally high exchange capacity, Reillex 402 may be preferable for some batch processes.

## Preparation of the Resin

**Aqueous Solutions** - Reillex 425 and 402 resins, as supplied, contain approximately 55% and 10% water, respectively. For best results, it is necessary to condition (equilibrate) the resin in the starting solvent prior to use. Conditioning is accomplished by soaking the resin in the solvent for several hours using either a column or a batch technique. When using the batch technique, the time required for conditioning can be reduced by refluxing a stirred slurry of the resin.

**Nonaqueous Solutions** - For applications involving water immiscible solvents, the resins can be dried in a standard drying oven at 80-110° C. Drying can also be accomplished by adding toluene (or other immiscible solvent having a water azeotrope) to the wet resin followed by distillation of the water/toluene azeotrope. The toluene can be removed by further drying or solvent exchange. The dry resin should be conditioned in the new solvent as described above.

## Preparation of the Column

To pack a column with Reillex 425 resin, slurry the resin with water (or other solvent) and pour the slurry into the column. The volume of the slurry should be at least twice the final volume of the settled resin bed. The finished column should not be filled more than about halfway.

## Backwashing

Before the first use, and after each cycle, the column should be backwashed (upflow, 100% expansion) to classify the beads and to remove insoluble material and air pockets.

## Operation of the Column

A normal cycle for operation of a Reillex column is a three-step process:

### 1. Exhaustion

The metal-containing solution is passed through the column while monitoring the effluent for breakthrough of the metal. When the metal concentration in the effluent reaches the influent level, the column is exhausted and ready for regeneration. Continuous, and more efficient service is achieved by using a three-column (Merry-Go-Round) system. Two columns are in service while the third column is regenerating.

### 2. Elution

**a. Competing ions** - adsorbed metal ions are eluted by introducing ions which compete for resin binding sites (eq.1). Since Reillex Resins have a very strong affinity for protons, H<sub>2</sub>SO<sub>4</sub> or other mineral acids are generally used for this purpose.

**b. Neutralization** - when the metal is adsorbed as the anionic complex, neutralization with a base destroys the interaction and frees the metal ion for elution (eq.2).

### 3. Regeneration

The column is rinsed to remove residual eluant, then backwashed and equilibrated with the starting solvent. The column is now ready to use again.

## suggested column operating conditions for Reillex 425

pH range	1-14
maximum temperature	225°C (air)
	260°C (absence of air)
minimum bed depth	24 inches
service flow rate	0.5 to 5 gpm/ft <sup>3</sup>
regenerants inorganic, aqueous	3-15% HC <sub>1</sub> or H <sub>2</sub> SO <sub>4</sub>
	2-8% NaOH
	1-4% NH <sub>3</sub>
regenerant flow rate	0.5 to 3 gpm/ft <sup>3</sup>
rinse flow rate	0.5 to 3 gpm/ft <sup>3</sup>

*CAUTION: Reillex Resin, like other organic resins, may swell or contract when changing solvent, pH, or ionic strength. This swelling can cause extreme pressures to build up, which may result in the violent rupture of a glass column. If significant swelling is expected, the solvent should be admitted upflow into an expanded bed.*



# applications

## how to put Reillex Resins to work

You may already have an application in mind for Reillex Resins. If so, our resin applications team is eager to help you develop it.

### Vanadium Extraction from Salt-Roast Leach Solutions

Vanadium (V) oxide concentrates are produced by an alkaline leaching process known as salt-roasting. A variety of feed sources (e.g., iron ores, uranium-vanadium ores, assorted slags and spent catalysts) provide salt-roast leach solutions which vary in vanadium content from trace amounts to 30-50 g  $V_2O_5/L$ . The oxy-vanadium ions can be extracted from the leachate with Reillex 425 resin.

A simulated salt-roast leach solution was prepared by dissolving light brown  $V_2O_5$  (3.0g) in 5% aq  $Na_2CO_3$  (1.0 L, 80°C) followed by addition of  $H_2SO_4$  to achieve pH 3.5. The solution was passed through a glass column (1 cm x 44 cm) packed with Reillex 425 (4.5 g, dry basis). Upon completion, the resin was rinsed with 50 mL  $H_2O$ . The vanadium-loaded beads were removed from the column, stirred with 5%  $Na_2CO_3$  (100 mL, 80°C) then filtered. Addition of  $NH_4Cl$  (10.0 g) to the filtrate precipitated 1.1g white  $NH_4VO_3$ .

### Recovery of Chromic Acid Wastes

Toxic chromic acid wastes from industrial cooling towers and electroplating operations are a major pollution problem. Chromate levels can be reduced by using a column of Reillex 425 resin. The chromates are stripped from the column by 5% aq NaOH. A glass column (2.5 cm x 120 cm) was packed with Reillex 425 resin (72 g, dry weight). A  $CrO_3$  solution (1.25 g/L) was passed through it. Chromium was completely removed until breakthrough (10% of influent concentration) occurred after 7.5 L. The adsorbed chromate was stripped with aqueous 5% NaOH solution.

### Recovery of Precious Metals

Reillex Resins are particularly effective for concentrating dilute solutions of precious metals. Reillex 425 resin is recommended for noble metal extraction from ores and wastes, the separation of precious metals from base metals, and the recovery of spent catalysts. The absorbed metals can be eluted from the column with a solution of thiourea. Alternatively, the resin can be burned to leave the metal in a very pure and concentrated form.

### Recovery of Palladium from Electroplating Wastewater

Simulated palladium electroplating wastewater (2 L) containing 0.2%  $PdCl_2$ , 0.1%  $NH_4Cl$  and 0.05% HCl was filtered through a column of Reillex 425 resin (15.0 g, dry weight) to completely remove the metal. The palladium was quantitatively stripped from the column with 75 mL of 5% aq thiourea/1% HCl solution.

### Recovery of Hydroformylation Catalysts

An interesting recovery system for cobalt hydroformylation catalyst has been described by Moffat.<sup>6</sup> Under an  $H_2/CO$  atmosphere, the active hydroformylation species  $HCo(CO)_4$  is strongly adsorbed by a poly-(vinylpyridine) resin. The metal is readily released under a CO atmosphere as  $Co_2(CO)_8$ .

The recycle system separates the catalyst from the product stream and recycles it without consuming chemical reagents or producing unwanted products.

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