The Chromatographic Separation of Copper from simulated Zinc and Nickel Targets

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Abstract

Copper-64 and Copper-67 radionuclides form a perfect theranostic pair that can be used to image and treat cancer and other disease. ^{64}Cu and ^{67}Cu are commonly produced, respectively, by irradiation of zinc or nickel targets. Chromatographic techniques using ion exchange and extraction chromatographic (EXC) materials are commonly used to efficiently separate metal ions from complex mixtures. EXC materials containing oxime, amide, and amine extractants have been shown to exhibit selectivity for copper over nickel, zinc, and other metal ions that may be present as impurities in target materials or as byproducts from target irradiation. EXC resins produced with N,N,N′,N′-tetraoctyl-3,6-dioxaoctane diamide (DOODA), tertiary amine, and aldoxime extractants and silica stationary phases with strong cation exchange (SCX) and weak cation exchange (CM) functional groups were evaluated in batch contact experiments for the retention Cu, Zn, Ni and other main group and transitional metal ions from hydrochloric, nitric, and sulfuric acid. Based on batch retention data, column chromatographic methods for the separation of copper from simulated zinc and nickel targets were developed to obtain high purity copper in small volumes of dilute hydrochloric acid or acetate buffer.

Production and Target Dissolution

Diagnostic:
^{64}Cu ($t_{1/2}$ = 12.7004 hours)
β^+ (β_{max} = 653.1 keV) 17.52%
β^- (β_{max} = 579.4 keV) 38.48%
ε 44.00%
γ (511 keV, 35.04%)

^{64}Ni(p,n)^{64}Cu
^{64}Zn(n,p)^{64}Cu

Therapeutic:
^{67}Cu ($t_{1/2}$ = 61.83 hours)
β^- (β_{max} = 561.7 keV)
γ (184.577 keV, 48.7%)

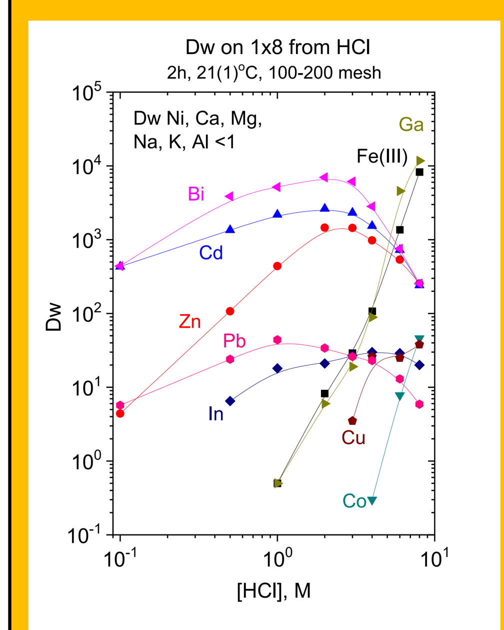
^{68}Zn(γ,p)^{67}Cu
^{70}Zn(p,α)^{67}Cu
^{68}Zn(p,2p)^{67}Cu
^{67}Zn(n,p)^{67}Cu
^{nat}Zn(p,2pxn)^{67}Cu

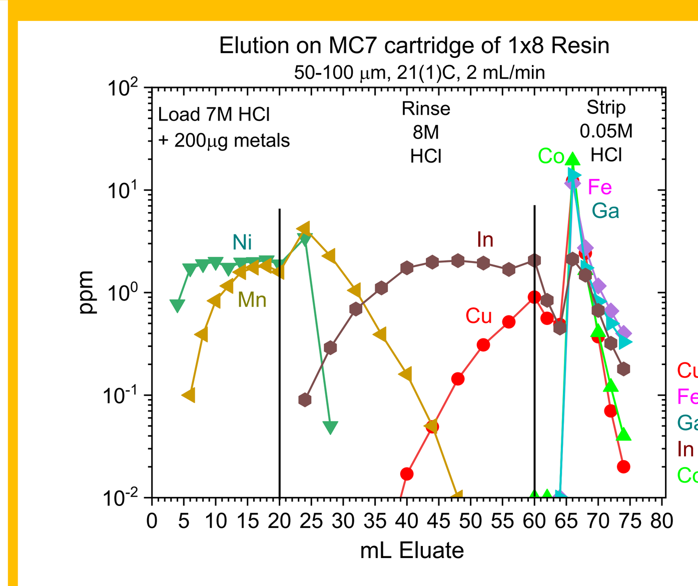
Nickel stable isotopes: 58(68.08%), 60(26.22%), 61(1.14%), 62(3.63%), 64(0.93%)
Zinc stable isotopes: 64(48.6%), 66(27.9%), 67(4.1%), 68(18.8%), 70(0.6%)

^{64}Cu and ^{67}Cu are produced by proton, neutron, or photon bombardment of isotopically enriched nickel or zinc targets. Zn metal and ZnO are easily dissolved in HCl, with very high purity Zn requiring elevated temperature. Nickel metal is dissolved with HNO_3 or HCl at elevated temperature. A few drops of 30% H_2O_2 can help dissolve any passivating NiO.

$Zn_{(s)} + 2HCl \longrightarrow Zn^{2+}_{(aq)} + 2Cl^-_{(aq)} + H_{2(g)}$
$ZnO_{(s)} + 2HCl \longrightarrow Zn^{2+}_{(aq)} + 2Cl^-_{(aq)} + H_2O$
$Ni(s) + 2HNO_3 \longrightarrow Ni^{2+}_{(aq)} + 2Cl^-_{(aq)} + H_{2(g)}$

1x8

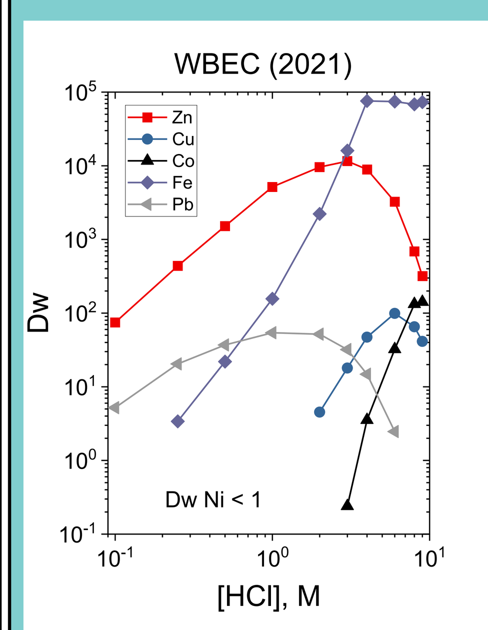


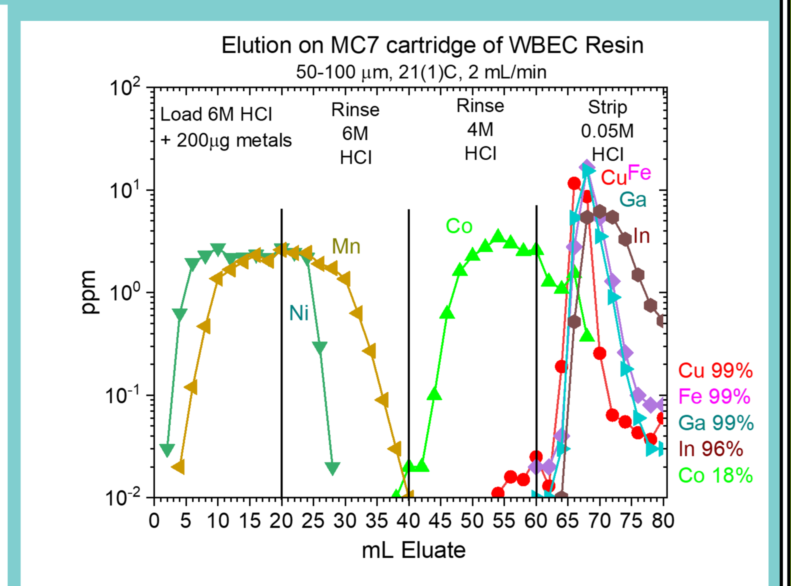


Cu can be separated on strong based anion exchange resins, such as Dowex 1x8 or MP-1 from Ni targets dissolved in high concentrations of HCl.

However the kd values for Cu are somewhat low, requiring relatively large columns and elution volumes. Also, separation factors for Co(II), Fe(III), and Ga(III) are poor, requiring secondary columns for purification. The strong base anion exchange columns do make good secondary purification columns due to their covalently bound functional groups, which limits organic impurities in the final purified Cu, and because the Cu can be recovered from 1x8 in dilute HCl or buffer solution.

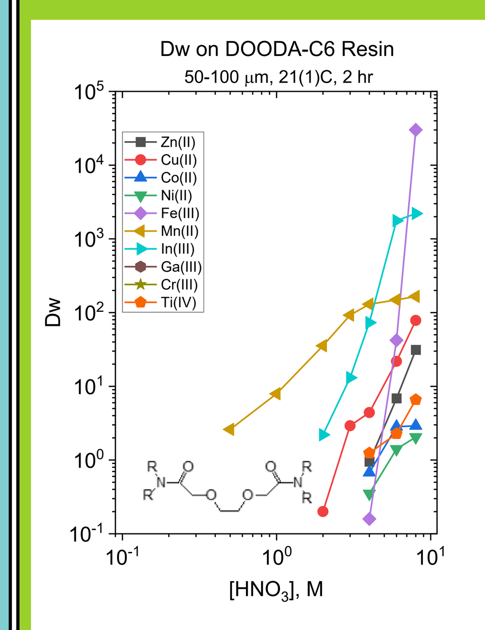
WBEC

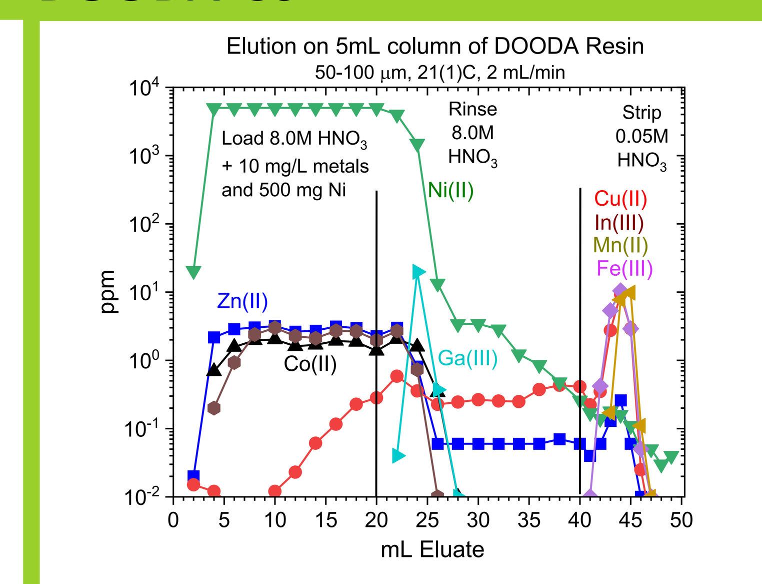


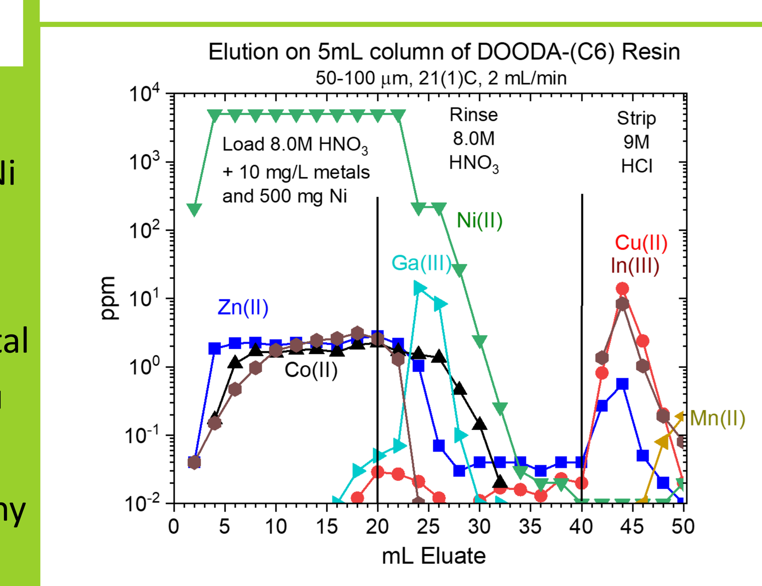


Weak base extraction chromatography resin (WBEC), based on tertiary amine extractants sorbed onto an inert substrate perform similarly to the 1x8 Resin, separating Cu from Ni targets dissolved in high concentrations of HCl. The uptake values for Cu are still somewhat low, requiring relatively large columns and elution volumes and selectivity of Fe, Zn and Ga is poor. However, separation factors for Co(II), are better than for 1x8. The WBEC columns also make good secondary purification columns allowing recovery of Cu in dilute HCl or buffer solution.

DOODA-C6

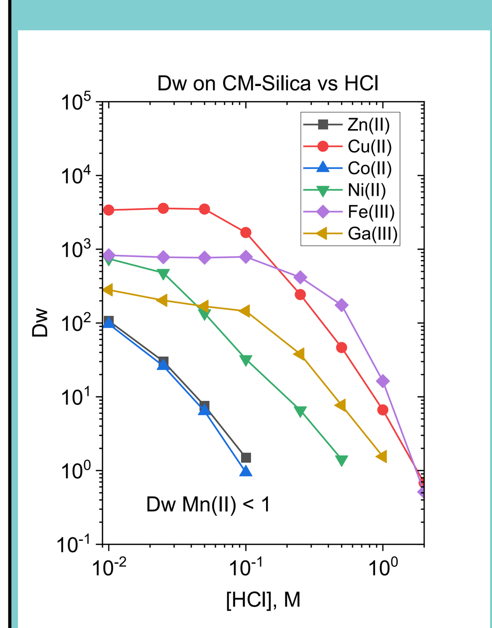


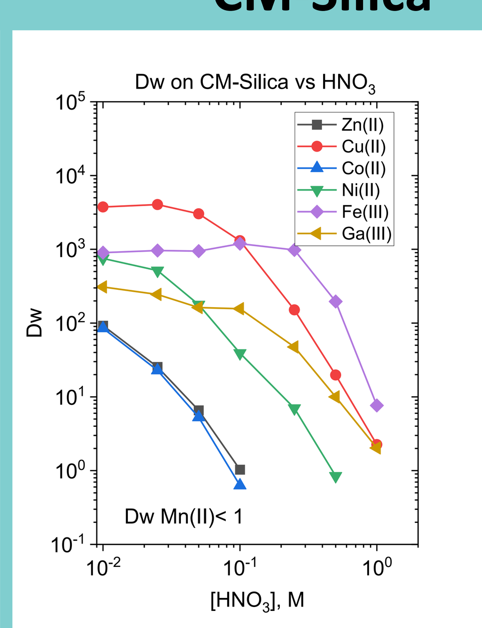


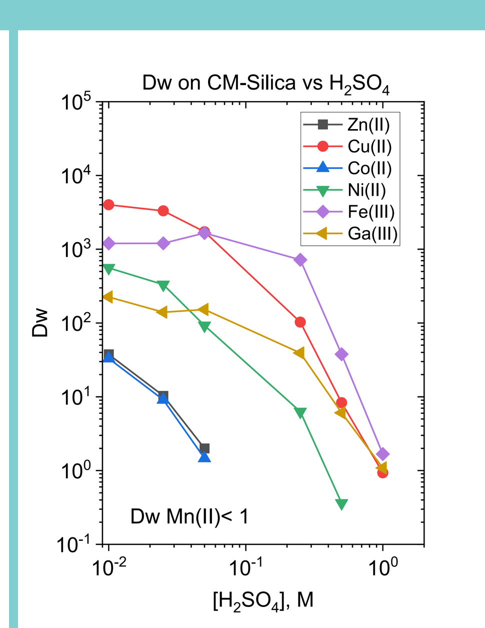


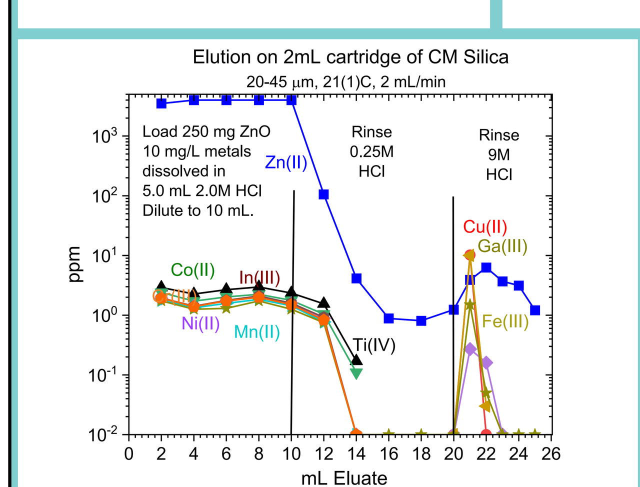
The DOODA-C6 resin offers good selectivity for Cu over Ni and other transition metals from HNO_3, allowing separation of Cu from Ni metal targets dissolved in HNO_3. Cu can be recovered from the DOODA-C6 in dilute HNO_3, any concentration of HCl, or acetate buffer. Pairing with a secondary column helps to remove HNO_3 and provide additional purification from Fe(III), Mn(II), In(III), Ga(III) and Zn(II).

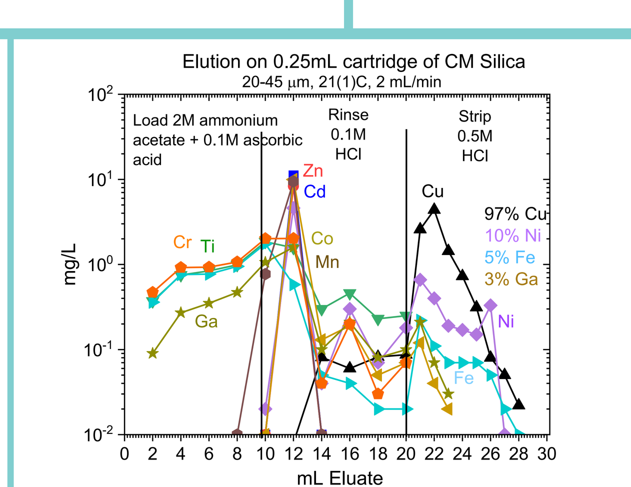
CM-Silica





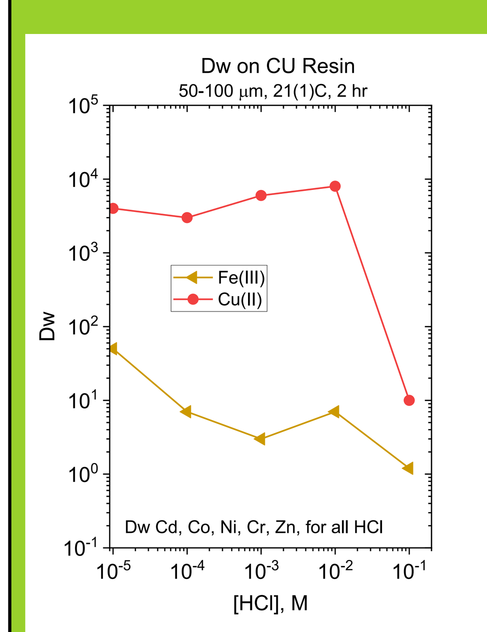


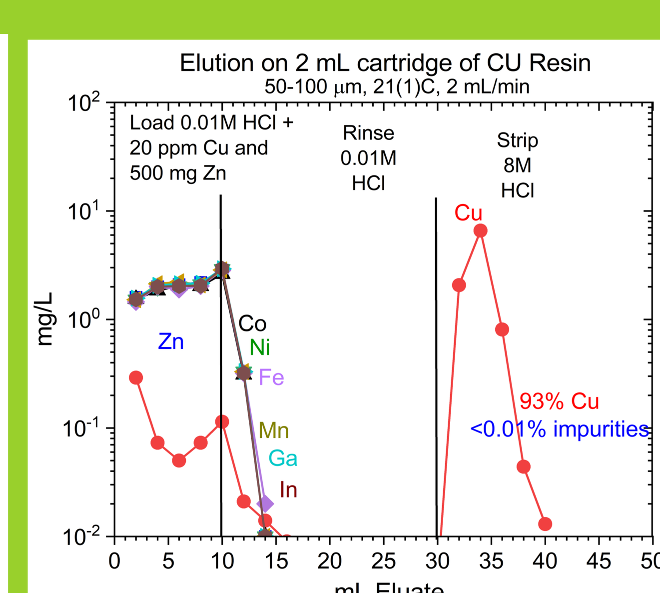


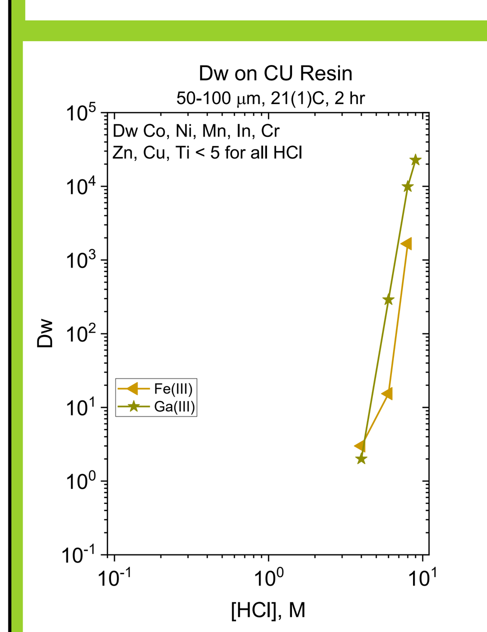


CM-silica offers selectivity of Cu over Zn for separation from Zn or ZnO targets dissolved with HCl, HNO_3, or H_2SO_4. Cu can be recovered from the CM with 0.50 - 9M HCl. Additionally, the CM silica can be used as a secondary column, loaded in acetate buffer to remove HNO_3, concentrate Cu, and provide additional purification from Zn, Cd, Co, Mn, Fe(III) and Ga(II).

CU Resin







CU resin offers great selectivity for Cu over Zn and Ni, making it equally functional for separation from Ni and Zn targets. However, Cu loading must be performed at pH 2-5, requiring evaporation following target dissolution or pH adjustment. Both approaches risk formation of insoluble oxides that can affect final Cu purity. Cu resin can also be paired with 1x8 or WBEC to provide additional purification and concentration of Cu and recovery in dilute HCl or buffer.

Conclusions/Future Work

A series of extraction chromatography and ion exchange resins have been evaluated for the separation of Cu from simulated Zn and Ni targets, identifying two column systems that provide high purity Cu in a small volume of dilute HCl. Future work will include evaluating the performance of the separation methods with larger Zn and Ni target masses and finding partners to test the methods using irradiated Zn and Ni targets.

References

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3) http://www.lnhb.fr/nuclides/Cu-64_tables.pdf

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5) https://www.triskem-international.com/scripts/files/59d1f4fc2c2091.54193347/tki6_en_binderonline_1.pdf

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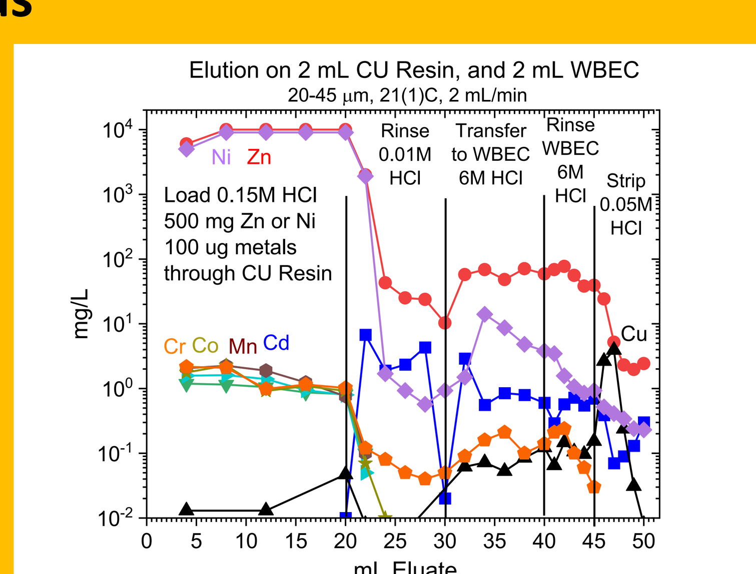
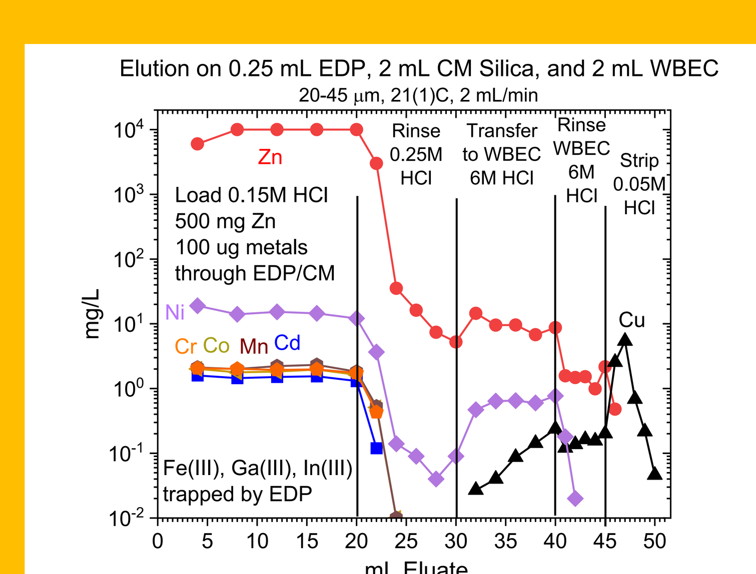
Dual column methods

500 mg of Ni or Zn metal or ZnO is dissolved in HCl, evaporated to dryness and redissolved in 20 mL of 0.01M HCl. The solution is loaded onto a 2 mL cartridge of CU Resin, rinsed and transferred to WBEC with 6M HCl. The Cu is recovered from WBEC with dilute HCl. Ni/Zn impurities in the final Cu fraction may be due to ZnO/NiO formed during evaporation and not dissolved in the 0.01M HCl.



500 mg of Zn metal or ZnO is dissolved in a small excess of HCl and diluted to 20 mL with 0.1M HCl. The solution is loaded onto a stack of 0.25 mL EDP Resin + 2 mL cartridge of CM-silica, rinsed with 0.25M HCl, and transferred to WBEC with 6M HCl. The Cu is recovered from WBEC with dilute HCl. Fe, In, and Ga are trapped by EDP resin guard column, with additional Fe and Ga contamination trapped on the CM-silica.



EDP Resin = H2DEH[EDP] on a methyacrylate support, a geminal diphosphonic acid with 2 carbons spacing the P=O groups.

500 mg of Ni metal is dissolved in HNO_3 and diluted to 20 mL of 8M HNO_3. The solution is loaded onto a 2 mL cartridge of DOODA-C6 Resin, rinsed with 8M HNO_3 and transferred to CM-silica with 10 mL of 2M ammonium acetate. The CM-silica is rinsed with 0.1M HCl to remove Mn, Cd, Zn, and Cu is recovered in 3-5 mL of 0.5M HCl. Ascorbic acid cannot be added to the ammonium acetate buffer to reduce Fe to Fe(II) as this will also reduce Cu(II) to Cu(I).