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**Distribution Ratios on Dowex 50W
Resins of Metals Leached
in the Caron Nickel
Recovery Process**

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ABSTRACT

Pressurized ion exchange on Dowex 50W-X8 and 50W-X12 resins was investigated using elution techniques to determine distribution ratios for copper, nickel, and cobalt complexes contained in ammonium carbonate solution, a mixture which approximates the waste liquor from the Caron nickel recovery process. Results were determined for different feed concentrations, as well as for different concentrations and pH values of the ammonium carbonate eluant. Distribution ratios were compared with those previously obtained from a continuous annular chromatographic system. Separation of copper and nickel was not conclusively observed at any of the conditions examined.

Contents

	<u>Page</u>
1. Summary	1
2. Introduction	1
3. Apparatus and Procedure	2
3.1 Equipment	2
3.2 Preparation of Solutions	2
4. Results and Discussion	4
5. Conclusions	12
6. Recommendations	12
7. Acknowledgment	13
8. Appendix	14
8.1 Sample Calculations	14
8.2 Original Data	16
8.3 Location of Original Data	17
8.4 Nomenclature	17
8.5 Literature Referenced	17
8.6 Literature Surveyed	18

1. SUMMARY

Pressurized ion exchange was used to study the separation of nickel, cobalt, and copper complexes present in the waste liquor of the Caron process. The chromatographic elution of these solutes was investigated on Dowex 50W-X8 and 50W-X12 resins with ammonium carbonate eluant. The conditions examined included eluant concentrations ranging from 0.5 to 2.0 M, eluant pH values ranging from 7.8 to 10.0, and feed solutions containing equal concentrations by weight of 1.0 or 4.0 g/l of each metallic species. Although cobalt complexes readily separated from the remaining species, separation of the nickel and copper complexes was not conclusively observed. Distribution ratios for all species declined with increased eluant concentrations. In addition, nickel and copper distribution ratios increased greatly with increased eluant pH.

Results were generally unaffected by feed concentration, which suggested linear adsorption isotherms over the range of compositions examined. The resin with a higher degree of crosslinking generally had a greater affinity for the copper-nickel complexes. Corresponding distribution ratios obtained previously on a continuous annular chromatograph were slightly lower than those obtained from the column-elution technique employed in this study, which may possibly be due to minor hydrodynamic differences between each apparatus.

2. INTRODUCTION

In the winning of nickel, one of the major processes is the Caron process, in which nickel is leached from reduced, roasted, laterite ores using an ammonium carbonate solution (4). The resulting liquor, containing nickel, cobalt, and other impurities (including copper) is heated under oxidizing conditions, and most of the nickel is precipitated. The remaining stream still contains quantities of nickel as well as cobalt and trace metal impurities. One suggestion for purification of the remaining nickel is to use ion exchange (9). Hurst (7) showed that such a process does indeed separate the copper and cobalt species.

Ion exchange chromatography has had widespread use in analytical work. The industrial applications have been limited to biochemical separations in pharmaceutical manufacture and water treatment. Ion exchange is usually performed in batch mode, and scale-up of exchange columns leads to decreased separation efficiency (6). The Chemical Technology Division of ORNL has been working on a continuous annular chromatograph (CAC) that may alleviate these problems (4, 11). Much work has been done in characterizing the CAC using a nickel-cobalt-copper system similar to the Caron process liquor on Dowex 50W-X8 cation exchange resin. To further the development of the CAC for this process, independently measured distribution ratios were needed. The purpose of this study was to determine the distribution ratios of the nickel-cobalt-copper system for the Dowex 50W-X8 and 50W-X12 exchange resins, while

varying the pH, eluant concentration, and species concentration. These ratios were obtained from data collected on a pressurized column chromatograph and were then compared with results from the CAC.

3. APPARATUS AND PROCEDURE

3.1 Equipment

Distribution ratios were determined on a pressurized column chromatograph. A schematic of the experimental system is shown in Fig. 1. Two column systems were operated in parallel. The eluant solution was forced through the column by a positive displacement pump (a Milton Roy minipump) with adjustable stroke length. The columns were packed with one of two cationic exchange resins, Dowex 50W-X8 and 50W-X12 (8 and 12% crosslinked, respectively). Both resins were 200-400 mesh (37-63 μm) in NH_4^+ form. The ends were packed with 0.3-0.4 mm. glass beads to prevent spreading of the resin bed. Samples of the metal species were injected by a syringe in a pulse by flushing a 1.0- cm^3 sample loop in a six-way valve onto the top of the column. The effluent from the column was monitored on a Visible-light setting, with solute absorbance recorded on a Variar chart recorder. A graduated cylinder was used to confirm these elution volumes. All connections in the system were 0.16-cm-OD x 0.28-cm-ID Teflon tubing. The column dimensions were initially 60 x 1.3-cm-ID. Due to the time required for a run (~ 2 h), the size of the column was decreased to 30 x 1.3-cm ID, then to 15 x 1.3 cm ID. Since copper-nickel separation was apparently not achieved, a 30 x 0.65-cm column was used in the final runs.

3.2 Preparation of Solutions

The metal species samples roughly simulated the process stream from the Caron nickel process, although they were much richer in copper. The actual process produces an ammonium carbonate solution containing nickel, three species of cobalt, and small amounts of copper. The simulated solutions contained equal solute concentrations on a mass basis of either 1.0 g/l (0.0170 gmol Ni/l, 0.015 gmol Cu/l, and 0.016 gmol/l for each of the Co species) or 4.0 g/l. Solutions were prepared from metallic copper and nickel dissolved in ammonium carbonate and from the nitrate salts of cobalt tetrammine $[\text{Co}(\text{NH}_3)_4\text{CO}_3]^+$ and cobalt pentammine $[\text{Co}(\text{NH}_3)_5\text{CO}_3]^+$. Each of these components elutes as a brightly colored band which is easily visible. The pH of the resulting solution was 9.7.

Ammonium carbonate eluant solutions were varied in concentration from 0.5 to 2.0 M. The pH of these solutions was increased to 10 by addition of concentrated ammonium hydroxide (58 wt %), and was decreased to a pH of 7.8 by bubbling carbon dioxide through the solution for several hours. The resins were regenerated by flushing the columns with several pore volumes of fresh eluant.

The elution volume for each species was measured, and a distribution ratio for each was calculated using the formula $K_D = (V_r - V_0)/(V_t - V_0)$, where K_D is the distribution ratio, V_r is the elution volume, V_t is the total bed volume, and V_0 is the void volume. Runs were made at constant superficial velocities of 3.2-15 cm/min (135-445 kPa) with feeds for which cationic contents were usually less than 5% of the bed capacity. Void volumes were determined by the relation $V_0 = 0.4 V_t$, for which the validity was confirmed experimentally using Blue Dextran tracer.

4. RESULTS AND DISCUSSION

The values of K_D are presented in Table 1 for the 8 and 12% resins, where the missing data points are due to time constraints. Since in the course of our work two columns were run in parallel while being monitored by a single spectrophotometer, only one point could be obtained when two peaks were eluted simultaneously. In addition, some runs were conducted to specifically monitor the copper-nickel absorption wavelengths. Because in most cases only three absorption peaks were clearly observed, we assumed that the copper and nickel were not separated and were thus eluted simultaneously.

A close scrutiny of the data at eluant pH values of 8.0 disclosed the possibility of a small Ni peak under a large, strongly absorbed Co-1 (cobalt tetrammine) peak. This phenomenon was observed on three consecutive runs in the 30 x 0.65-cm-ID column, two with the feed solution at 4 g/l of each metal, and one special run with the feed at 4 g/l for Ni and at 1 g/l for all other metals. The Ni peak was smaller since it was obscured by the Co-1 peak, which was four times larger. The nickel peak would be further obscured by the loss of resolution observed at greater superficial velocities. It is not surprising that the apparent Ni peaks were not observed under these conditions.

In the final runs the pH of the eluant gradually increased to about 0.4 unit above its initial value of 8.0. Therefore the distribution ratios reported at an eluant pH of 8.0 were actually values for a slightly higher pH, where copper-nickel separation was less likely to occur. After the conclusion of this study, copper-nickel separation was observed using a smaller sample size, which decreased the pH change in the column (1).

The effect of pH on distribution is quite different for each metal species (Figs. 2 and 3). On both resins, K_D values for cobalt tetrammine (Co-1) pass through a maximum at pH 8.9. For the 12% resin, K_D values for cobalt pentammine (Co-2) increase slightly with pH. K_D values for nickel and copper show a marked decline with decreasing pH and actually elute before Co-1 at pH values of 8.4 or less. This behavior was confirmed by Hurst (7) for the 8% resin.

The reduced affinity of the nickel and copper species for the resin at lower pH may be explained by an association reaction in which bicar-

Table 1. Calculated Distribution Ratios on Dowex Resins

Dowex 50W-X8									
pH	Eluant Concentration (g/l)	Feed Concentration (g/l)				Distribution Ratios			
		Co-1	Co-2	Ni	Cu	K _{Co-1}	K _{Co-2}	K _{Ni}	K _{Cu}
10.0	2.0	4	4	4	4	3.9	6.3	14.6	14.6
10.0	0.5	4	4	4	4	8.1	21.1	62.0	62.0
10.0	2.0	1	1	1	1	5.3	8.9	20.8	20.8
10.0	2.0	1	1	1	1	5.3	5.3	23.2	23.2
10.0	0.5	1	1	1	1	18.0	62.1	135.3	135.3
8.9	2.0	4	4	4	4	1.5	4.7	7.3	7.3
8.9	0.5	4	4	4	4	15.5	44.7	35.5	35.5
8.9	2.0	1	1	1	1	4.1	6.5	8.2	8.2
8.9	0.5	1	1	1	1	16.7	39.7	35.6	35.6
8.9	0.5	4	4	4	4	15.5	44.7	35.5	35.5
8.9	0.5	1	1	1	1	16.7	39.7	35.6	35.6
8.9	1.0	4	4	4	4	7.1	13.5	10.8	10.8
8.9	1.0	1	1	1	1	8.1	15.1	10.0	10.0
8.9	1.0	1	1	1	1	*	*	12.4	12.4
8.0	1.0	4	4	4	4	7.6	19.7	3.3	3.3
8.0	1.0	4	4	4	4	*	*	3.5	3.5
8.0	0.5	4	4	4	4	12.1	48.3	5.3	5.3
8.0	1.0	1	1	1	1	12.9	45.5	2.1	5.1
8.0	1.0	1	1	1	1	7.9	21.5	3.5	3.5
8.0	0.5	1	1	1	1	15.7	20.8	6.0	6.0
8.0	0.5	1	1	1	1	7.9	20.6	2.0	3.4
8.0	0.5	1	1	1	1	12.7	47.4	5.1	5.1
Dowex 50W-X12									
10.0	2.0	4	4	4	4	3.9	6.6	18.4	18.4
10.0	0.5	4	4	4	4	13.6	66.7	165.2	165.2
10.0	2.0	1	1	1	1	4.8	8.6	25.9	25.9
10.0	0.5	1	1	1	1	12.9	97.0	188.6	188.6
8.9	2.0	4	4	4	4	4.5	6.1	6.7	6.7
8.9	0.5	4	4	4	4	14.7	45.9	33.0	33.0
8.9	2.0	1	1	1	1	4.5	6.1	6.5	6.5
8.9	0.5	1	1	1	1	7.4	16.9	14.3	14.3
8.9	0.5	1	1	1	1	15.4	*	50.1	50.1
8.9	2.0	1	1	1	1	4.1	*	8.3	8.3
8.9	0.5	4	4	4	4	14.7	*	33.0	33.0
8.9	2.0	4	4	4	4	3.8	*	7.9	7.9
10.0	0.5	1	1	1	1	14.4	*	256.8	256.8
10.0	0.5	4	4	4	4	7.7	*	78.6	78.6
8.9	0.5	4	4	4	4	14.7	45.9	33.0	33.0
8.9	0.5	1	1	1	1	18.1	61.9	43.2	43.2
8.9	1.0	4	4	4	4	7.5	17.1	14.5	14.5
8.9	1.0	4	4	4	4	6.4	14.6	14.4	14.4
8.9	1.0	1	1	1	1	7.4	16.2	15.0	15.0
8.0	1.0	4	4	4	4	6.5	23.0	3.3	3.3
8.0	0.5	4	4	4	4	10.9	62.9	5.3	5.3
8.0	1.0	1	1	1	1	7.6	25.0	3.8	3.8
8.0	1.0	1	1	1	1	*	*	3.2	6.0
8.0	0.5	1	1	1	1	11.3	53.3	6.1	18.4
8.0	0.5	1	1	1	1	12.4	47.7	1.1	2.1

Eluant concentration = 0.5 M
 $(\text{NH}_4)_2\text{CO}_3$
 Feed concentration for each species
 = 1 g/l

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VARIATION OF K_D WITH pH
 12% RESIN

DATE	DRAWN BY	FILE NO.	FIG.
10-20-79	MEM	CEPS-X-301	2

Eluant concentration = 0.5 M $(\text{NH}_4)_2\text{CO}_3$
 Feed concentration of each species
 = 1 g/l

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VARIAION OF K_D WITH pH
 8% RESIN

DATE 10-20-73	DRAWN BY MEM	FILE NO. CEPS-X-301	FIG. 3
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bonate ions (formed from increased CO_2 levels dissolved in water) replace water attached to the nickel complex, thereby reducing the overall charge of the complex according to (2):

Increased eluant concentrations lead to sharply reduced distribution ratios for both resins at all conditions examined (Figs. 4 and 5). This observed behavior is predicted by the mass-action model for the species equilibrium, which predicts that the distribution ratio decreases proportionally with the square of the eluant concentration. However, the second-order dependence is not absolute since the activity coefficient of the eluant changes with molarity and pH. These results are also corroborated by the literature (7).

Changes in feed concentration did not significantly change distribution ratios for either resin (Table 1). This indicates a linear adsorption isotherm within the tested range of species concentrations. The exception to this behavior can be noted at pH values of 10, where small nonlinearities would be most noticeable since the distribution ratios are rather large (~10-260). Since the column loading was calculated to be no greater than 4% of the bed capacity, indicating that overloading of the column did not occur, the cause of this discrepancy remains unexplained.

Comparison of the 8 and 12% columns are of special interest (Figs. 2 and 3, or Figs. 4 and 5). Values of copper and nickel distribution ratios for the 12% resin were noticeably higher at high eluant pH values, where the data were less scattered. On the other hand, the cobalt species were not affected significantly by the amount of crosslinking. The increased species selectivity of the 12% resin was shown previously for Cu and Ni (10) but is not understood fully at this time.

The chief advantage of the 12% resin lies in its decreased swelling, which facilitates industrial equipment design. However, higher crosslinking generally leads to poorer resolution of each species (8). The difference in band spreading between the resins may be most easily seen from the theoretical plate model. Column efficiencies, expressed as number of theoretical plates, may be determined by (10):

$$N = 8(V_r/W)^2 \quad (2)$$

where N is the number of theoretical plates, V_r is the elution volume of each species, and W is the bandwidth in volume units. For similar operating conditions, the 8 and 12% resins show plate ratios of 30-40% (see Table 2), indicating the resolution advantages of the 8% resin.

Eluant pH = 8.9

Feed concentration of each species
= 4.0 g/l

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EFFECT OF ELUANT CONCENTRATION ON K_D
8% RESIN

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FIG.
4

Eluant pH = 8.9

Feed concentration of each species
= 4.0 g/l

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EFFECT OF ELUANT CONCENTRATION ON K_D
12% RESIN

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FIG.
5

Table 2. Theoretical Plates Required for Nickel Separation

Eluant pH	Concentration of (NH ₄) ₂ CO ₃ Eluant (M)	Feed Concentration Ni:Co-1:Co-2:Cu (g metal/g)	Theoretical Plates 50W-X12	Plates 50W-X8
8.0	0.5	1:1:1:1	54	82
8.9	1.0	4:4:4:4	76	119
10.0	2.0	4:4:4:4	580	948

Superficial velocity of the eluant varied from 3.2 to 15.0 cm/min. However, since ion exchange equilibria are rapid, the flow rate has no effect on the peak eluant position or on the distribution ratios (10).

The range of distribution ratios [2M (NH₄)₂CO₃] were close to those observed by Hurst (7) for the 8% column. This confirms the accuracy of our experimental technique. However, when compared with the distribution ratios for the same Ni-Cu-Co system run on the CAC, several discrepancies were observed. Values of the distribution ratios obtained by Scott (Table 3) (11) are uniformly lower than those obtained by column experiments.

Table 3. Comparison of Column and CAC Distribution Ratios

[Eluant: 1 M (NH₄)₂CO₃; pH 7.8; 8% Resin]

	Ni	Co-1	Co-2	Source
K _D	2.92	4.70	11.62	Scott et al. on continuous annular chromatograph (11)
	3.98	5.57	14.80	Scott et al. on column chromatograph
	3.48	7.88	21.51	This study

This small but consistent variation may be attributable to difference in hydrodynamic properties of the two systems. For simplicity, the influence of hydrodynamic dispersion on chromatographic resolution has been ignored in our analysis, a treatment which is commonly used when examining adsorption of species from fluids passing through porous bodies. However, the open radial boundary for a CAC fluid element implies that lateral hydrodynamic dispersion is important in that system. This premise is supported by researchers examining dispersion in the absence of sorption. They have

shown that two-dimensional hydrodynamic dispersion must be considered when the lateral system boundary is not rigid [i.e., see (3)]. Differing lateral dispersion phenomena of the two systems, which will alter longitudinal dispersion and sorption behavior, would consistently create the type of second-order effect observed in Table 2.

Another difference between the column and CAC experiments was the apparent inability to separate Ni and Cu in the column experiments, while this separation was easily achieved on the CAC. The uncertainty noted above for the Cu and Ni data indicates the caution required in comparing the resolution potentials of each apparatus.

5. CONCLUSIONS

The following conclusions can be drawn for the system tested (copper, nickel, and two cobalt ammine complexes in ammonium carbonate solution):

1. The copper-nickel species distribution ratio was markedly decreased with decreasing pH. The two cobalt species had distribution ratios relatively independent at pH values ranging from 8 to 10.
2. Distribution ratios for all species declined with increased ammonium carbonate concentration.
3. For the most part, distribution coefficients were independent of species concentration.
4. The 12% resin generally showed greater selectivity for nickel species than did the 8% resin, but produced poorer resolution of species peaks.
5. Copper-nickel separation was not observed at eluant pH greater than 8.9. The separation observed at pH 8 was questionable due to uncertainties in experimental procedures.

6. RECOMMENDATIONS

1. Similar tests should be performed using different eluants, i.e., ammonium sulfate, ammonium bicarbonate, or ammonium chloride, as investigated in previous work (7).
2. A detailed analysis of the CAC should be performed to resolve differences in distribution ratios obtained on a column.
3. Other metal systems of industrial interest, zirconium-hafnium and aluminum-titanium-iron, should be analyzed.

4. The possible separation of the copper and nickel species should be examined in greater detail.

7. ACKNOWLEDGMENT

We thank Fred Hurst, John Begovich, Sam Clinton, and Ron Canon for their assistance during this study.

8. APPENDIX

8.1 Sample Calculations

A typical elution curve is given in Fig. 6. Calculation of distribution ratios proceeds directly from the peak elution volume (V_r), the bed volume (V_t), and the void volume of the bed (V_o) according to:

$$K_D = \frac{V_r - V_o}{V_t - V_o}$$

where

$$V_o = 0.4 V_t$$

For the nickel peak on the 8% resin when run at an eluant pH of 8.0, an eluant concentration of 1 M, and a feed concentration of 4 g/l, K_D becomes:

$$K_D = \frac{54 - 3.4}{8.5 - 3.4} = 9.9$$

The number of theoretical plates is reported to be given by (10):

$$N = 8\left(\frac{V_r}{W}\right)^2$$

where V_r is defined above, and W is the band width measured at 37% of the maximum peak height (H_1/e).

For the same run as above:

$$N = 8\left(\frac{54}{14}\right)^2 = 119$$

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TYPICAL RUN

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8.2 Original Data

Dowex 50W-X12

pH	Eluant (M)	Feed (g/l)	V_r (cm ³)				V_o (cm ³)
			Co-1	Co-2	Ni	Cu	
10.0	2.0	4.0	89.4	142	375	375	13.1
10.0	0.5	4.0	159	434	1556	1556	13.1
10.0	2.0	1.0	36.4	61	175	175	4.4
10.0	0.5	1.0	99	99	1699	1699	4.4
8.9	2.0	4.0	29.4	29.4	56.4	56.4	4.4
8.9	2.0	1.0	31.4	31.4	59.4	59.4	4.4
8.9	0.5	4.0	104.4	104.4	300	300	4.4
8.9	0.5	1.0	106.4	106.4	335.4	335.4	4.4
8.0	1.0	4.0	47.4	156	26.4	26.4	4.4
8.0	1.0	1.0	54.4	169	29.4	29.4	4.4
8.0	0.5	4.0	76	419	39.4	39.4	4.4
8.0	0.5	1.0	86	319	18.4	11.4	4.4
8.0	0.5	1.0	79	326	44.4	126	4.4
8.0	1.0	1.0			76.4	133	13.3
8.0	1.0	1.0 (Ni = 4)			81.4	135.4	13.3
8.9	0.5	1.0	41.4	89.4	76.4	76.4	3.4
8.9	1.0	4.0	34.4	76.4	74.4	74.4	3.4
8.9	2.0	4.0	26.4	34.4	37.4	37.4	3.4
8.9	2.0	1.0	26.4	34.4	36.4	36.4	3.4
8.0	0.5	1.0	210	787	137	137	3.4
8.9	0.5	1.0	94.4	319	220	220	3.4
8.9	0.5	4.0	77.4	234	169	169	3.4
10.0	0.5	4.0	94	444	1094	1094	4.4
10.0	0.5	1.0	89.4	644	1249	1249	4.4

Dowex 50W-X8

10.0	2.0	4.0	93	141	306	306	13.5
10.0	0.5	4.0	177	441	1269	1269	13.5
10.0	2.0	1.0	39	39	177	177	4.4
10.0	0.5	1.0	123	414	897	897	4.4
8.9	2.0	4.0	14	35	52	52	4.4
8.9	2.0	1.0	31	47	58	58	4.4
8.9	0.5	1.0	114	266	239	239	4.4
8.0	1.0	4.0	54	134	26	26	4.4
8.0	1.0	1.0	56	146	27	27	3.4
8.0	0.5	1.0	88	317	38	38	11.8
8.9	0.5	4.0	106.5	299	194	194	4.4
8.0	0.5	4.0	84	323	39	39	4.4
8.0	0.5	1.0	57	140	27	18	3.4

Dowex 50W-X8 (continued)

pH	Eluant (M)	Feed (g/l)	V_r (cm ³)				V_o (cm ³)
			Co-1	Co-2	Ni	Cu	
8.0	1.0	1.0	86	307	34	15	4.4
10.0	2.0	1.0	39	63	141	141	4.4
8.0	1.0	4.0			74	74	11.8
8.0	1.0	1.0			51	108	11.8
8.0	0.5	1.0	289	379	117	117	11.8
8.9	1.0	1.0			199	199	3.2
8.9	1.0	4.0	38	67	54	54	3.2
8.9	1.0	1.0	41	75	59	59	3.2

8.3 Location of Original Data

The original data are found in Databook A-9176-G, pp. 1-71, on file at the MIT School of Chemical Engineering Practice, Bldg. 1505, ORNL.

8.4 Nomenclature

H_i	maximum height of the eluant peak, arbitrary units
H_i/e	height to measured band width, arbitrary units
K_D	distribution ratio defined in Sect. 3.2, dimensionless
V_r	elution volume at peak, cm ³
V_o	void volume of the bed, cm ³
V_t	total volume of the bed, cm ³
W	width of elution peak at H_i/e , cm ³

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