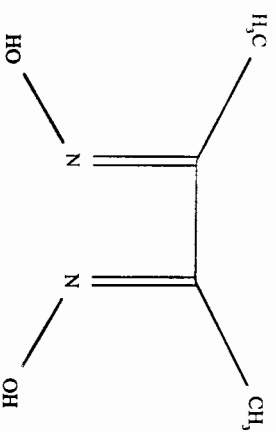


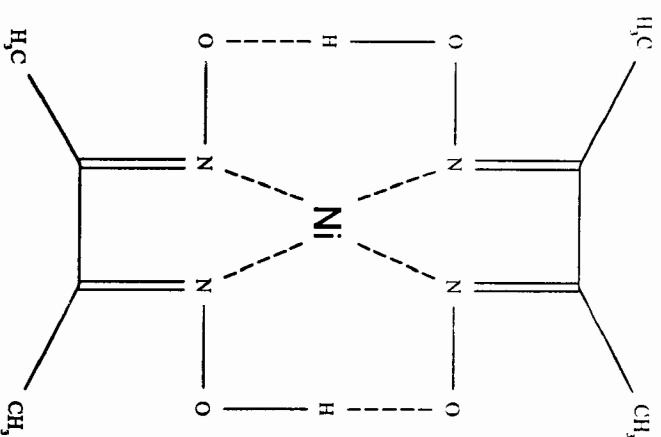
New Sample Preparation Schemes for Nickel and Tritium Analysis

Michael J. Fern

Eichrom Industries, Inc.



DMG

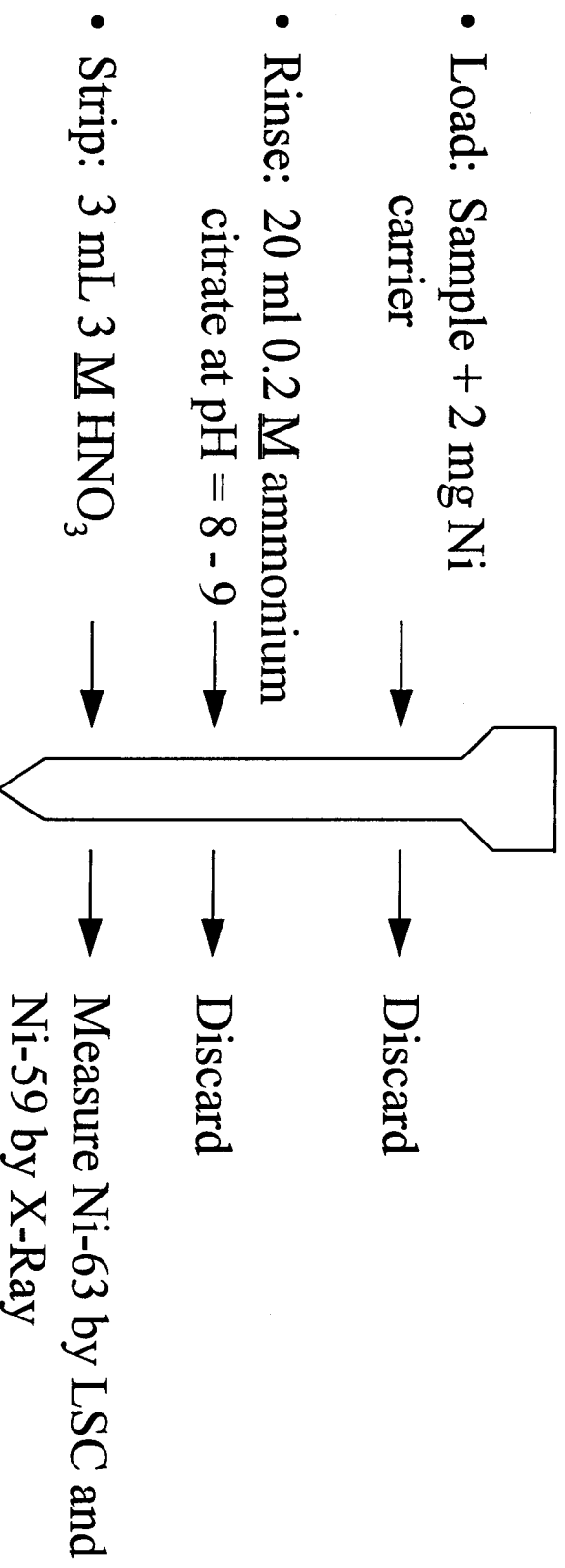


Ni-DMG Complex

Physical Characteristics of Eichrom's Nickel Resin

- 11% (w/w) DMG loading
- Resin Density ~ 0.25 g/mL
- Working Capacity ~ 3 mg Ni

Nuclear Power Plant Nickel Procedure (cont.)



Eichrom

INDUSTRIES, INC.

COMPARATIVE NI-63 ACTIVITY ($\mu\text{Ci}/\text{unit}$) DETERMINATIONS

<u>SAMPLE TYPE</u>	<u>STANDARD METHOD</u>	<u>NICKEL COLUMN</u>	<u>RATIO</u>
TL/HS Tank	2.30 E-6	2.38 E-6	0.97
Lab Waste Tank	2.66 E-6	2.49 E-6	1.08
WECT Tank	4.31 E-6	4.17 E-6	1.03
NI-63 Spike	5.07 E-4	5.35 E-4	0.95
NI-59 Spike	1.00 E-2	1.07 E-2	0.93
DAW Smears	4.18 E-2	4.63 E-2	0.90
Radwaste Filter	7.40 E-1	7.79 E-1	0.95
RWCU Resin	1.83 E+0	2.05 E+0	0.89

Decontamination Factors RWCU Resin Sample

Cr-51	>37,000
Mn-54	270,000
Co-58	110,000
Co-60	113,000
Nb-95	13,700
Cs-134	>9,000
Cs-137	58,000

MISCELLANEOUS OBSERVATIONS
ON EICHIROM NICKEL COLUMN PERFORMANCE

<u>PARAMETER</u>	<u>STANDARD METHOD</u>	<u>COLUMN METHOD</u>
Chemical Yield	63 ± 9%	90 ± 14
LLD (50 ml/30 min)	1.2 E-7	6.5 E-8
Analysis Time:		
Overall	12 Hours	4 Hours
Hands On	8 Hours	2 Hours
LSC Cocktail	Mixed Waste	_____

Commercial Laboratory Nickel Procedure

- Sample + 2 mg Ni Carrier

- Evaporate



Little or No Residue



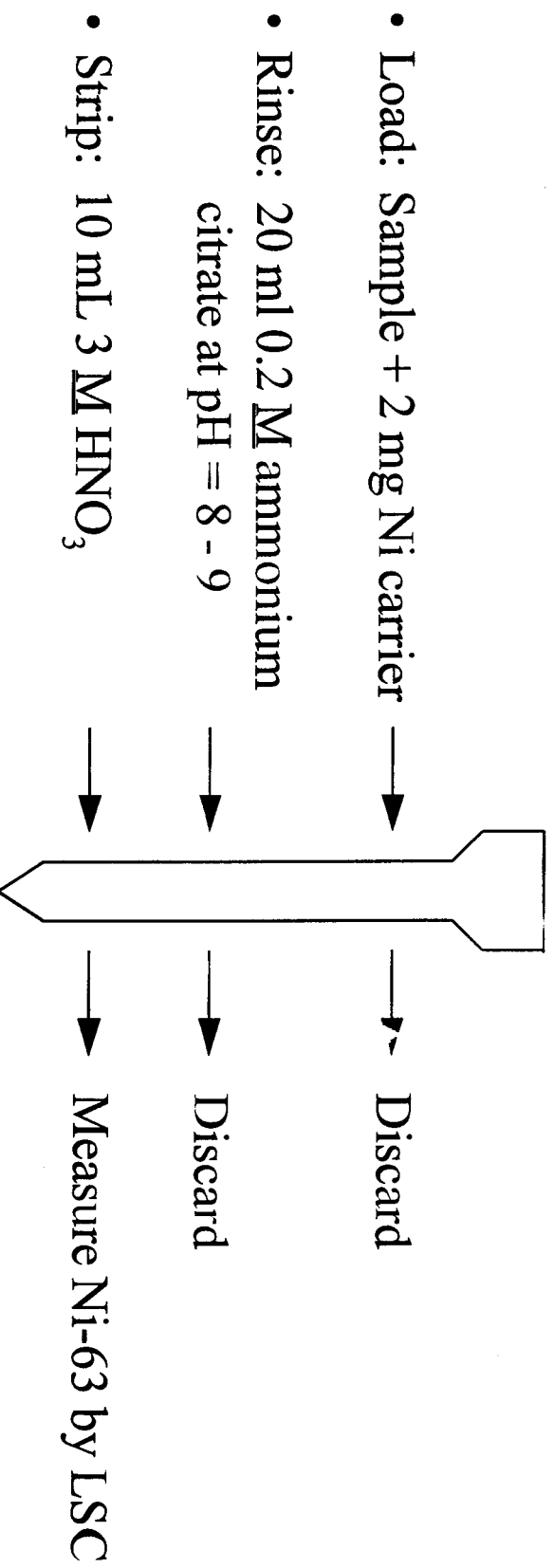
TRU Resin Fe Separation

Large Residue



Anion Exchange 1 x 8 column

Commercial Laboratory Nickel Procedure (cont.)



Summary of Ni-63 Results Using EIChrom's Ni Column at Commercial Lab

ID	Chem. Yield	Results	Error	Rad. Chem. Yield	Comments
	%	dpm/sample	dpm/sample	%	
Blank 1	91.60	0.114	1.309		No Fe Cleanup
Spike 1	91.70	45.000	3.830	89.95%	No Fe Cleanup
Spike 2	92.70	9.910	1.827	97.60%	No Fe Cleanup
Blank 3	95.60	0.044	1.255		Ion Exchange Fe Cleanup
Spike 5	86.70	46.590	3.990	92.64%	Ion Exchange Fe Cleanup
Spike 6	83.80	8.772	1.899	86.48%	Ion Exchange Fe Cleanup
Blank 4	89.70	-1.428	1.256		TRU Fe Cleanup
Spike 7	88.20	46.800	3.981	93.31%	TRU Fe Cleanup
Spike 8	96.10	9.781	1.775	96.35%	TRU Fe Cleanup
Blank 5	86.30	-0.704	1.598		TRU Fe Cleanup
Blank 6	83.90	-0.210	1.664		TRU Fe Cleanup
Spike 9	84.80	46.181	3.947	92.10%	TRU Fe Cleanup
Spike 10	77.90	7.612	2.121	75.00%	TRU Fe Cleanup
Sample 1	78.60	-1.621	1.724		TRU Fe Cleanup
Sample 2	85.10	-1.221	1.603		TRU Fe Cleanup
Dup. 1	85.60	-1.695	1.574		TRU Fe Cleanup
Dup. 2	88.00	-0.702	1.566		TRU Fe Cleanup

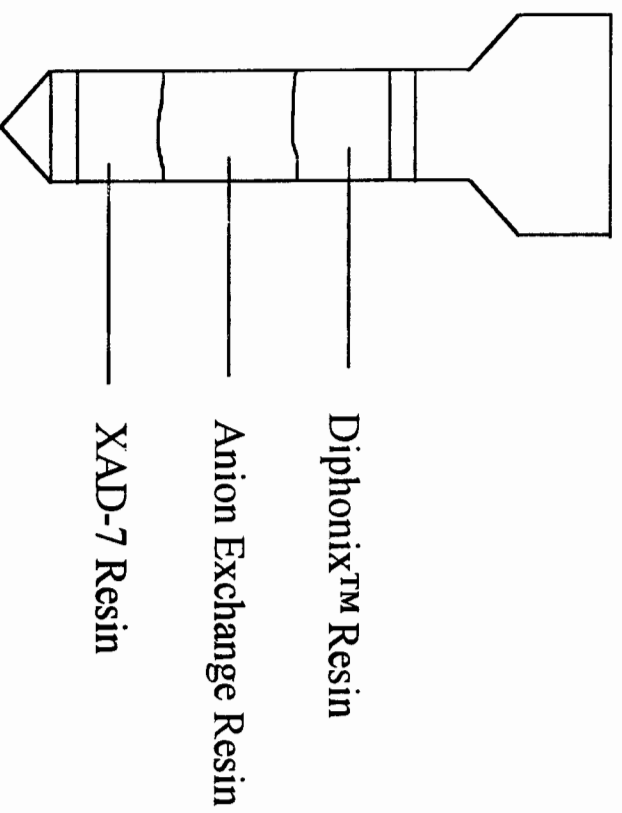
Miscellaneous Observations on Eichrom Nickel Column Performance

<u>Parameter</u>	<u>Std. Method</u>	<u>Column Method</u>
Chemical Yield	57 %	87%
LLD (200 mL/200 min)	7 pCi/L	5 pCi/L
Analysis:		
Elapsed	2 - 2 1/2 days	2 days

Nickel Resin: Summary

- In routine use in commercial labs and power plants
- Results equivalent to or better than the traditional procedure
- Lower cost and easier to use

Tritium Column Composition



Tritium Column

<u>Component</u>	<u>Target</u>	<u>Capacity</u>
Diphonix™ Resin	Cations	0.9 mEq
Anion Exchange Resin	Anions	0.9 mEq
XAD-7	Organic Molecules	50 mg

Tritium Column Spike Recoveries

<u>Sample Type</u>	<u>Sample Volume</u>	<u>³H Spike (Bq)</u>	<u>% Recovery</u>
Distilled Water	15 mL	97.2	96.4% (n=4)
Distilled Water	25 mL	20.2	87.6% (n=4)
Distilled Water	25 mL	19.5	94.9% (n=7)
Ground Water	25 mL	17.1	91.2% (n=5)
Sea Water	25 mL	4.0	90.0% (n=6)
Urine	25 mL	85.1	91.1% (n=4)

Mixed-Contaminant Solution

(16.9 Bq Total Activity)

<u>Isotope</u>	<u>Activity (Bq)</u>
Co-60	1.7
Cs-137	3.1
U-232	1.8
Sr/Y-90	4.8
Pb-210	3.9
Th-230	1.7

Contaminant Study Results

		Contaminant	
<u>Sample Type</u>	<u>Sample Vol.</u>	<u>³H Spike (Bq)</u>	<u>Spike (Bq)</u>
Distilled Water	25 ml	19.5	16.9
Distilled Water	25 ml	20.2	16.9
Sea Water	25 ml	4.0	16.9
			<MDA (n=2)
			<MDA (n=2)
			<MDA (n=4)

MDA: Minimum Detectable Amount

US Power Plant Test Results

³H Concentration Measured (Bq/L)

Sample Type	Distillation	Column
Surface Water-40	1.66 (±0.24) x 10 ²	1.92 (±0.25) x 10 ²
Surface Water-26	2.86 (±0.26) x 10 ²	2.99 (±0.27) x 10 ²
Groundwater-16	1.25 (±0.041) x 10 ³	1.26 (±0.041) x 10 ³
Groundwater-2C	1.73(±0.044) x 10 ³	1.66 (±0.044) x 10 ³
BWR-RCS	1.02 (±0.004) x 10 ⁵	1.01 (±0.004) x 10 ⁵
PWR-RCS	1.62 (±0.0) x 10 ⁷	1.52 (±0.0) x 10 ⁷

US Power Plant Testing

Decontamination from PWR Samples

<u>Isotope</u>	<u>Before column (Bq/L)</u>	<u>After column Bq/L)</u>
Cr-51	2,900	ND
Mn-54	518	ND
Co-58	4,740	ND
Fe-59	109	ND
Co-60	392	ND
Sn-113	230	ND
Nb-95	4,220	ND
Zr-95	2,210	ND
I-131	14,200	ND
Cs-134	1,129	ND
Cs-137	1,320	ND

ND: Not detected above background

US Power Plant Testing

Decontamination from BWR Samples

Isotope	Before column (Bq/L)	After column Bq/L)
Cr-51	1990	ND
Mn-54	5590	ND
Co-58	4960	ND
Co-60	5990	ND
Nb-95	116	ND
La-140	1550	ND
Ce-144	203	ND

ND: Not detected above background

French Fuel Reprocessing Plant

Test Results: ^3H Concentration (Bq/L)

<u>Sample</u>	<u>Distillation</u>	<u>Column</u>
1	2.05×10^7	2.04×10^7
2*	4.7×10^4	4.4×10^4
3*	1.8×10^6	2.1×10^6
4*	9.2×10^3	9.6×10^3
5*	6.4×10^3	6.5×10^3

*Samples 2 -5 had been acidified and ^3H fraction contained other β -emitters.

US Commercial Lab Results

Environmental Water Samples

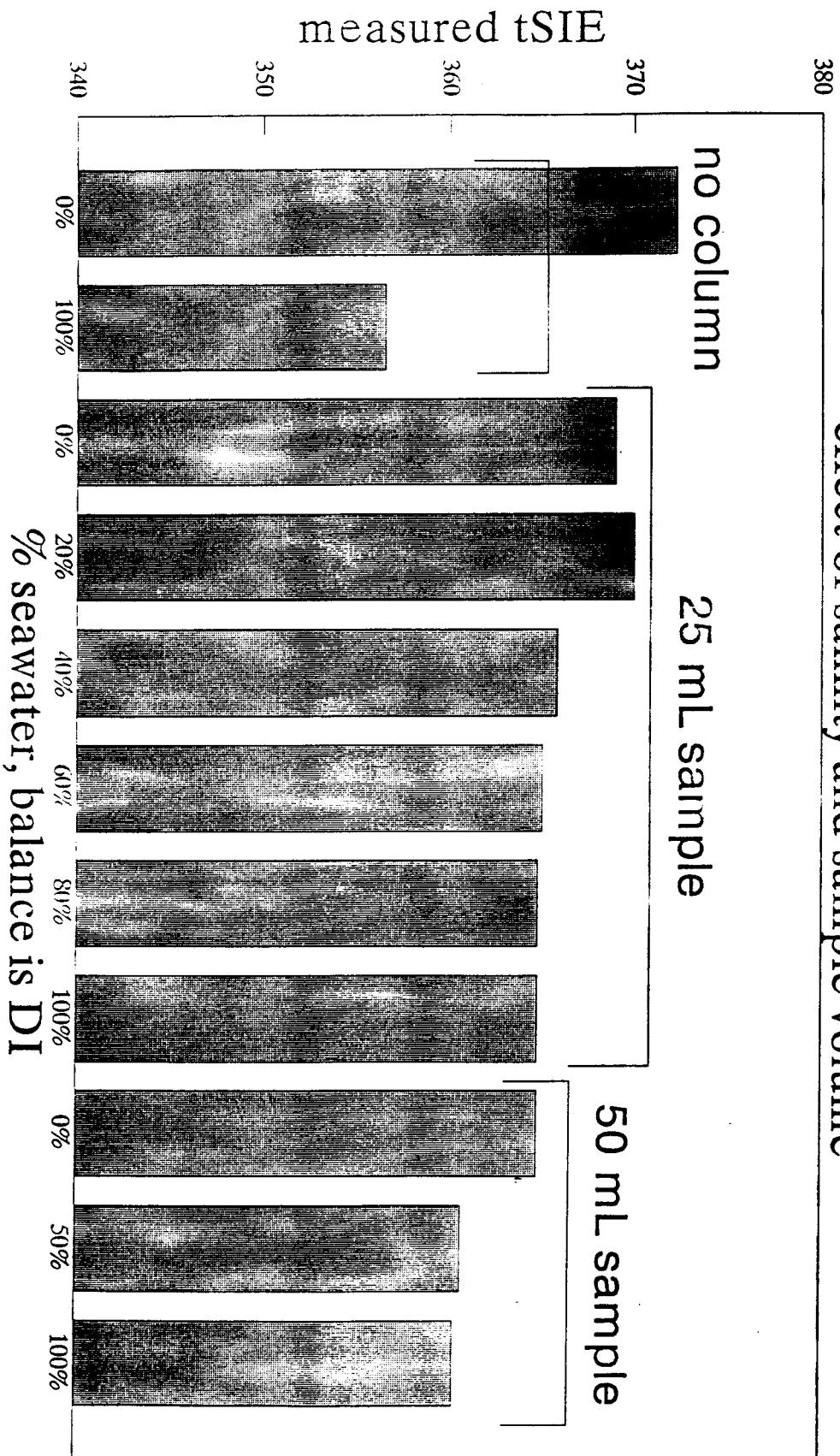
Tritium Concentration (Bq/L)

<u>Sample</u>	<u>Distillation</u>	<u>Column</u>
1	-0.096 ± 5.7	-0.546 ± 4.0
2	1.06 ± 6.03	-3.14 ± 4.0
3	243	221
4	242	230
5	-2.59 ± 7.77	2.22 ± 4.20
6	1.72 x 10 ⁶	1.75 x 10 ⁶
7	2.75 x 10 ⁵	2.71 x 10 ⁵
8	1.71 x 10 ⁶	1.64 x 10 ⁶
Spike 1	370*	352
Spike 2	1,990*	1,970

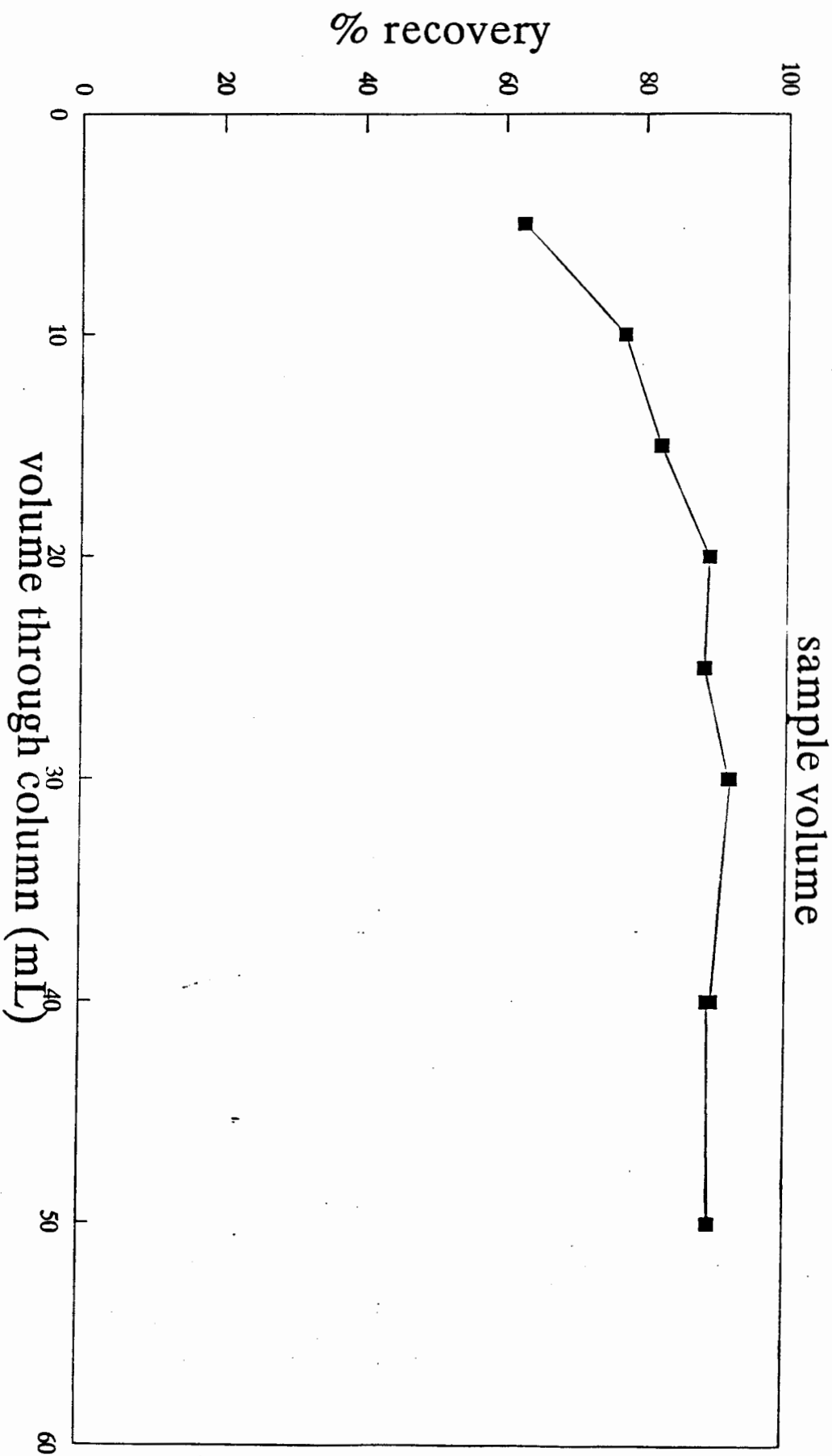
* Amount spiked

Tritium Column Performance

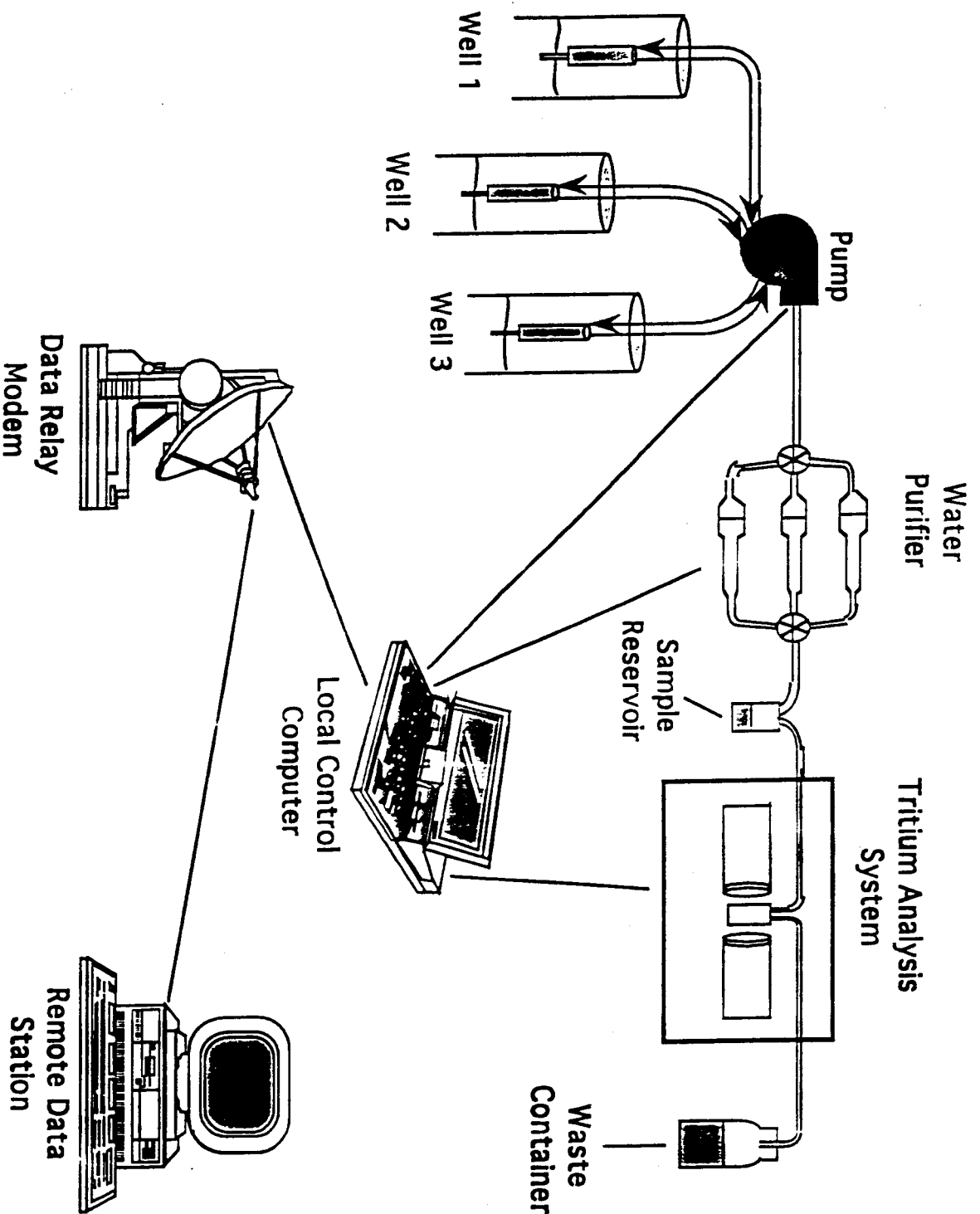
effect of salinity and sample volume

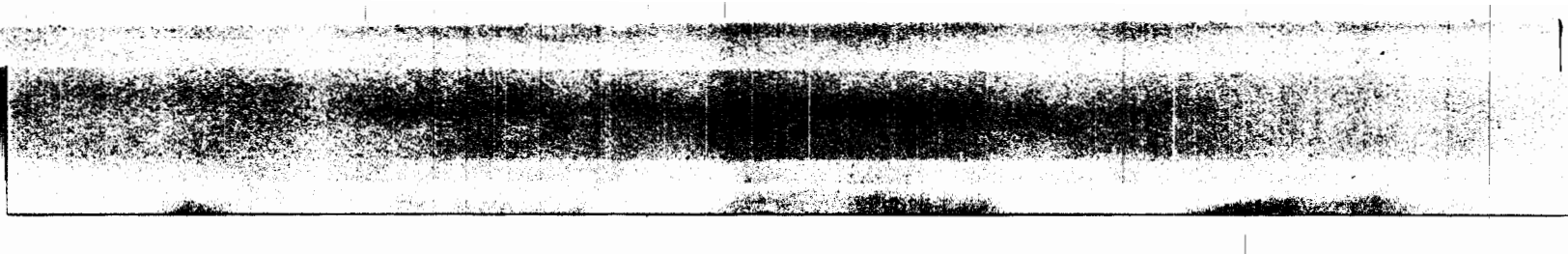


Tritium Column Performance



Remotely Operated Field Deployable Tritium Analysis System





Tritium Column: Summary

- In use worldwide
- Results equivalent to distillation
- Easier to use
- Possible to automate

Acknowledgments

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