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CONSTRUCTION AND OPERATION OF A CONTINUOUS ION-EXCHANGE PILOT PLANT USING FLUIDIZED-BED COLUMNS

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CONSTRUCTION AND OPERATION OF A CONTINUOUS ION-EXCHANGE PILOT PLANT USING FLUIDIZED-BED COLUMNS

by

P. Prud'homme* and B.H. Lucas**

SUMMARY

The development of new simplified ion-exchange equipment for recovering metals from solution is described. For many years fixed-bed ion exchange has been practised on clarified liquors. A more economical treatment was realized by constructing a continuous ion-exchange plant in which resin inventory is reduced and solution filtration and clarification stages eliminated. The equipment consists of a single-stage deep fluidized-bed extraction column coupled with moving-bed elution and wash columns for the extraction of different ions from mine water solutions and leach liquors. A procedure was developed to achieve the proper utilization and optimum operating conditions for this pilot-plant equipment.

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CONSTRUCTION ET OPERATION D'UNE INSTALLATION PILOTE D'ECHANGE D'IONS EN CONTINU, UTILISANT DES COLONNES A LIT FLUIDISE

par

P. Prud'homme* et B.H. Lucas**

SOMMAIRE

La mise au point d'un nouvel équipement simplifié d'échange d'ions pour la récupération de métaux en solution y est décrite. Depuis plusieurs années, l'échange d'ions utilisant un lit fixe a été exercé sur des liqueurs clarifiées. Un traitement plus économique a été réalisé par la construction d'une installation d'échange d'ions en continu; l'inventaire de la résine s'y trouve réduit et les étapes de filtration et de clarification de la solution y sont éliminées. L'équipement consiste en une colonne d'extraction d'un étage à profond lit fluidisé couplée à des colonnes d'élution et de lavage à lit mobile pour l'extraction de différents ions contenus dans des solutions d'eau de mine et des liqueurs de lixiviation. Une procédure pour parvenir à l'utilisation convenable des conditions optimales d'opération pour cette installation pilote a été mise au point.

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INTRODUCTION

The recovery of metals by fixed-bed ion exchange (IX) has been practised for many years. However, the operations were semi-continuous and could treat only clarified liquors. Substantial savings would be possible if the operation were made continuous, thus reducing resin inventory, and if the costly filtration and clarification steps could be eliminated. Work has also been in progress over the past twenty-five years on the recovery of metals from unfiltered ore leach slurries, heap leach liquors, and mine drainage waters containing small amounts of suspended solids. In the case of dilute waste solutions containing suspended matter and with high flow rates, filtration is usually prohibitively expensive.

Continuous ion exchange (CIX) using fluidized beds has made it possible to both considerably reduce resin inventory and to treat unclarified liquors and slurries. The subject has been reviewed on several occasions (1,2,3,4). Although fluidized beds in multi-stage columns are an improvement over previous resin-in-pulp treatment, they have a complicated internal structure and complex automatic control systems to move the resin stages.

M. Slater, of the University of Bradford, U.K., performed studies on multi-stage fluid beds, and concluded that the performance of single, deep fluidized beds of resin with continuous or semi-continuous flow of both phases would be worth investigating, particularly in the case of very dilute metal solutions (5). A pilot plant was designed and built at CANMET in a cooperative project with Dr. Slater. The use of deep fluidized beds of resin with a large height to diameter ratio for treating dilute solutions has the attraction of engineering simplicity. Extreme simplicity of equipment design is considered of paramount importance when treating, for example, mine waters at high flowrates.

In one case, a synthetic mine water containing 0.056 g/L (56 ppm) of copper and lesser amounts of Ni, Fe and Ca was treated in a deep fluidized IX bed, with Amberlite 200C resin to extract the metallic elements. This solution, treated in a continuous system at a flowrate of 44 $kL/h/m^2$, yielded a raffinate containing less than 0.02 mg/L (0.02 ppm) Cu. A 3M sodium chloride eluate containing 2.62 g/L of Cu was obtained which would be amenable to treatment by solvent extraction and IX for the separation of the metals, or alternatively, the metals could be bulk-precipitated. From this performance of the single-stage fluidized-bed IX unit, it was felt that a concentrated leach liquor such as a uranium acid liquor could also be treated successfully. A review of the processes investigated in the unit will be found in reference 6.

The purpose of this paper is to describe the equipment and to standardize the work procedure for operation of the single-stage deep fluidized-bed CIX pilot plant.

CONSTRUCTION OF COLUMNS

The general layout of the pilot plant is shown in Fig. 1. The extraction column is constructed of 51-mm diam Kimax glass tubes, 2.44 m long, standing vertically on a 0.91-m high support constructed of standard black angle iron, 51 mm x 0.032 mm in section, capable of adjustment up or down by 150 mm (Fig. 2). Above the extraction column is a sealed receiver for resin feed. The details of the extraction column construction and the sealed resin feed receiver are given in Fig. 3.

The support pipe to which the glass column is attached is made of standard black iron pipe, 38.1 mm in diam (Fig. 4). The pipe is connected to a flange bolted to the floor and is held in place by a clamp welded at the upper floor level. Holes are drilled 0.61 m apart through the pipe, to accommodate 3-prong clamps which hold the glass columns to the vertical pipe.

The elution column is 3.12 m high, made up of a 51-mm diam by 2.08-m Kimax glass tube surmounted by a 102-mm diam by 0.96-m section. Directly below and attached to the elution column is a 51-mm diam by 1.22-m glass wash column.

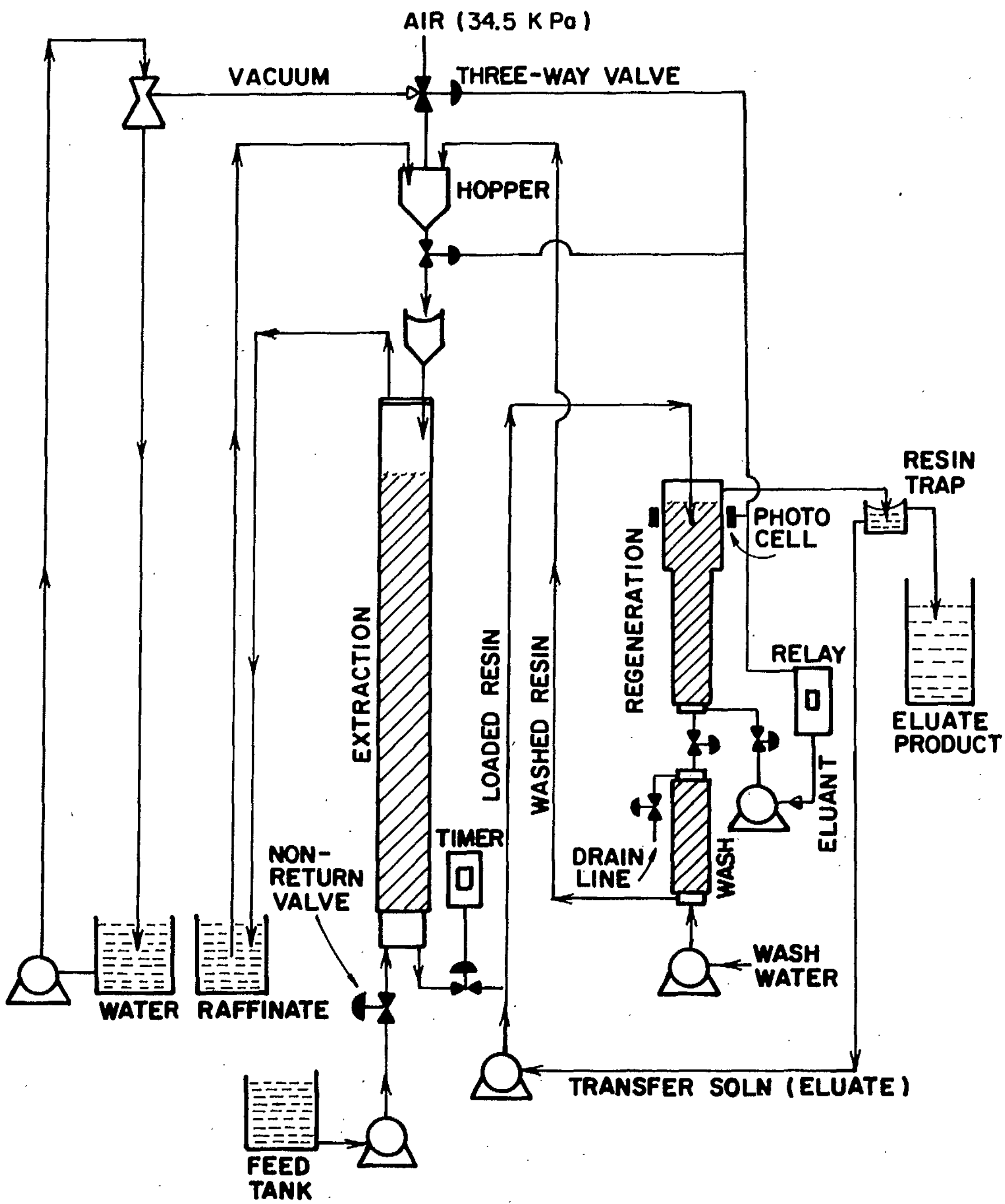


Fig. 1 - Continuous ion exchange pilot plant

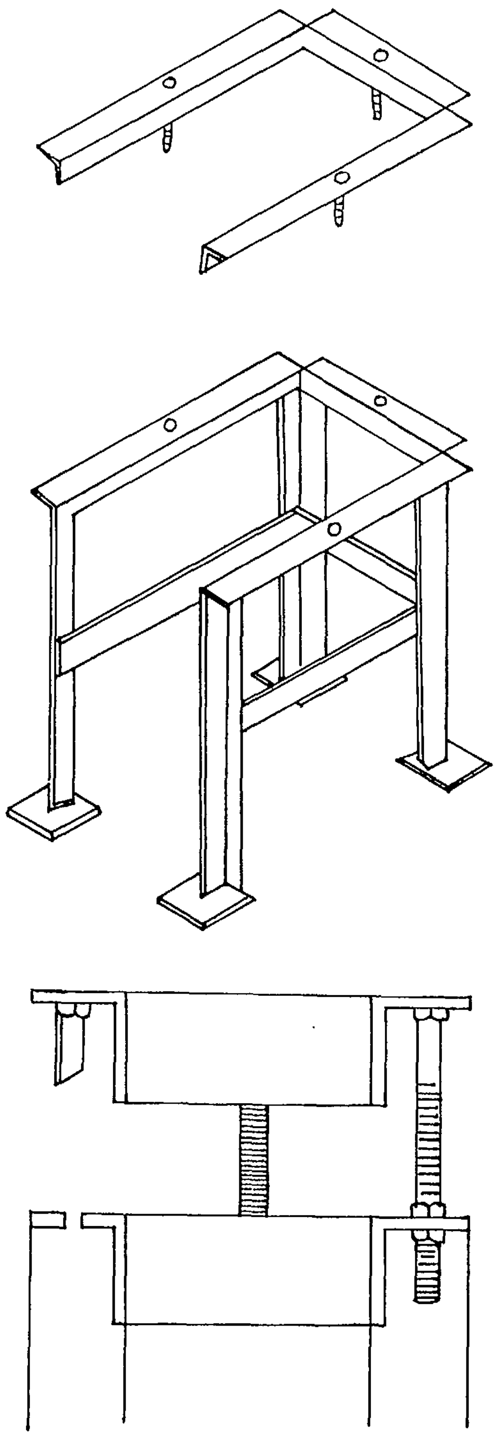


Fig. 2 - Column support stand

Details of the elution column are given in Fig. 5.

The support stand is placed beside the support pipe and the glass column is assembled, beginning with the bottom section and working upward. The glass pipe is connected by means of Kimax beaded process pipe couplings with Teflon inserts made for this purpose by the manufacturer. As excessive compression may cause the 51-mm diam glass to crack, care should be taken to keep the column perpendicular.

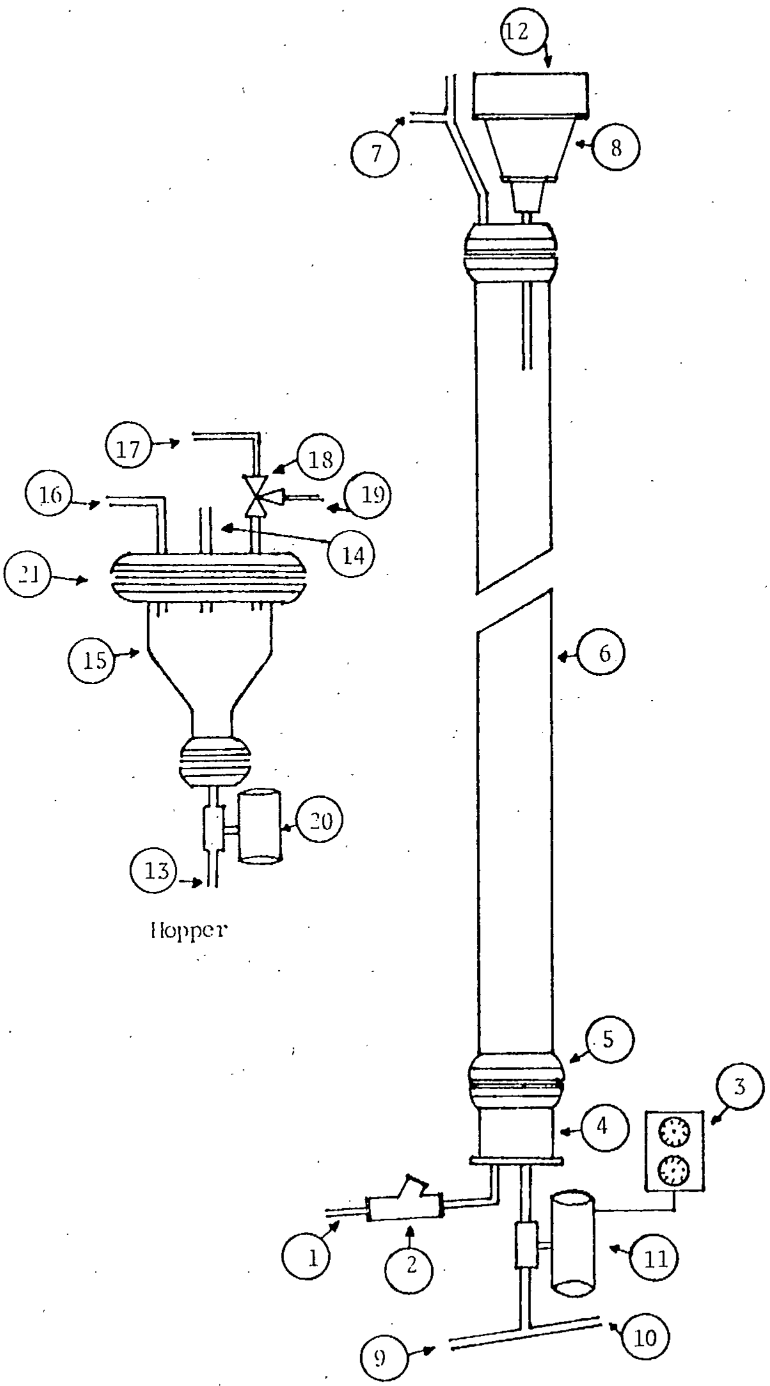
A complete list of equipment for the pilot plant unit including glass pieces, valves, pumps, timers, etc. is given in Appendix A.

OPERATION OF THE PILOT PLANT

The extraction column is operated with a metered upflow of solution, sufficient to maintain a measured volume of resin at a desired constant fluidized height. If pulp is used, the loaded resin plus pulp is transferred through a vibrating 147-µm screen where they are separated, the loaded resin entering the top of the regeneration column and the solid being collected in a tank. After a set time period, a calculated increment of loaded resin is discharged from the base of the column through an automatic ball valve controlled by a timer. The resin increment per minute is equal to V/60T where V = volume of resin in the extraction column and T = resin retention time in hours, given by the breakthrough point in a static run. Eluate solution is used to transfer the loaded resin to the top of the elution column.

Water is metered into the base of the wash column and flows upward into the elution column where it meets the eluant and dilutes it to a desired strength. The state of the resin in these columns is only slightly expanded. A photoelectric cell, with a light source placed on the 102-mm section of the elution column, maintains the desired height. Upon interruption of the light source by transfer of the loaded resin, a similar amount of resin is removed from the base of the wash column. Resin moves down the elution column through a 19-mm diam pipe directly into the wash column. This is a convenient arrangement of wash and elution cycles for pilot-plant studies.

The resin feed receiver vessel above the



(1) liquid feed inlet
(2) non-return 12.7 mm (1/2 in.) valve
(3) timer
(4) lucite or polyethylene section
(5) 50.8 mm (2 in.) and 152.4 mm (6 in.) coupling
(6) 50.8 mm (2 in.) x 2.44 m (96 in.) pipe
(7) raffinate outlet
(8) plastic receiver (funnel)
(9) transfer solution inlet
(10) loaded resin outlet
(11) 12.7 mm (1/2 in.) automatic ball valve
(12) washed resin inlet
(13) hopper discharge line
(14) washed resin inlet
(15) hopper 152.4 mm (6 in.) x 50.8 mm (2 in.) reducer
(16) rinse water
(17) vacuum line
(18) 3-way solenoid valve
(19) compressed air 34.5 KPa (5 psig)
(20) 12.7 mm (1/2 in.) automatic ball valve
(21) 50.8 mm (2 in.) and 152.4 mm (6 in.) coupling

Fig. 3 - Extraction column and hopper

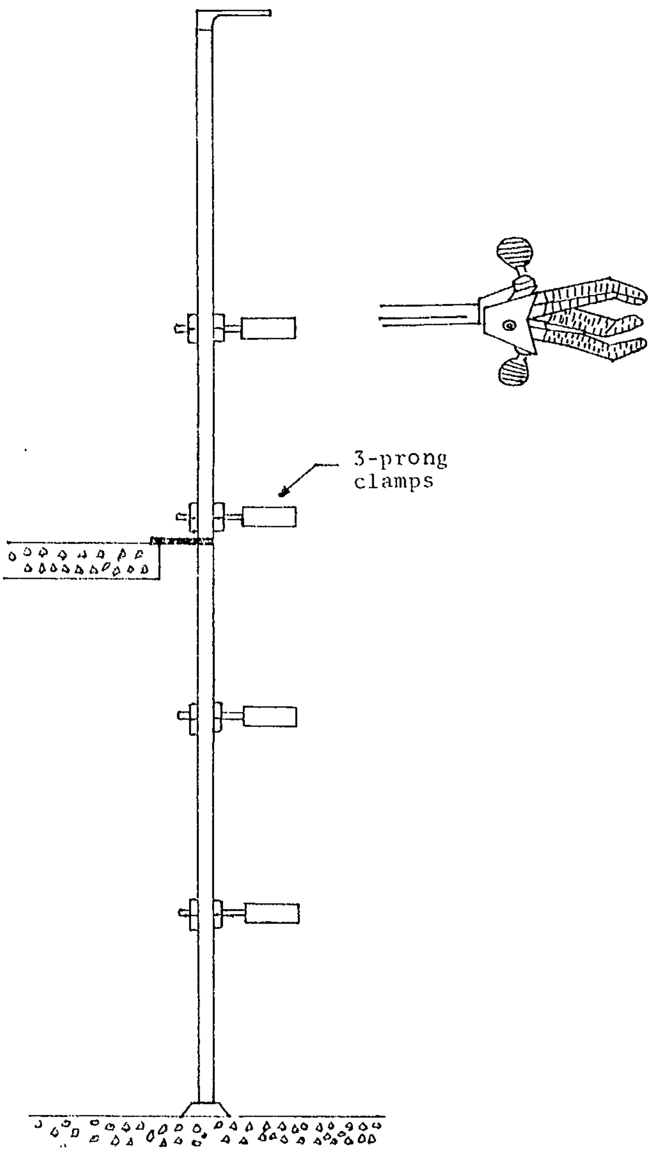


Fig. 4 - Column support pipe

extraction column is a sealed unit to which compressed air, vacuum and water can be applied. A three-way solenoid valve provides compressed air or vacuum. The photoelectric cell actuates the three-way valve which allows a vacuum to transport resin feed and water to the receiver vessel until the resin level in the elution column reached a predetermined set point. A timer regulates the volume of resin removed. The resin is then transferred to the extraction column by activation of a solenoid valve on the discharge where air pressure at 136 kPa and rinse water are used to flush the resin.

PRELIMINARY TESTWORK BEFORE CONTINUOUS OPERATION

Before starting any pilot-plant CIX investigation, the ion-exchange process must be known or developed from bench-scale tests which will indicate the pH of the feed solution, the eluant, the resin and its pre-equilibrated form. The next step is to run a series of extraction column fluidized resin bed static tests with no resin transfer, to establish operating conditions for CIX, i.e., to ascertain the feed liquor flowrate and resin bed retention time necessary to obtain the desired effluent quality.

The conditions for continuous operation are found from static tests run at different voidages, e.g., 0.5 to 0.8, where voidage, a function of the flowrate, is given by the formula:

$$\frac{1 - e}{0.63} = \frac{h_o}{h}$$

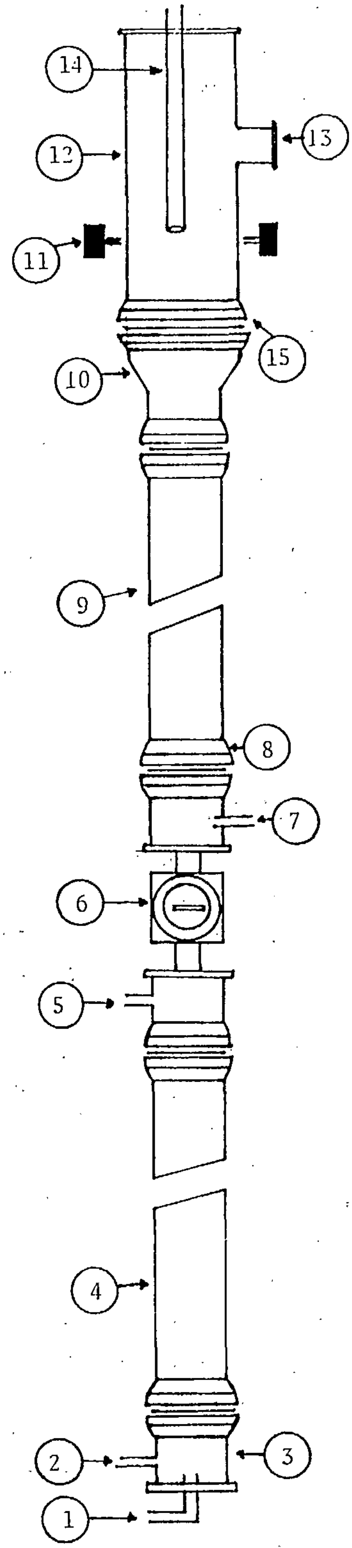
where e = voidage

h_o = settled resin height in m

h = expanded resin height in m (constant for each static run)

Feed liquor is passed through the resin at a rate necessary to maintain the desired expanded bed height, and samples of the effluent are taken. The analyses indicate the quality of the effluent being produced during the run until breakthrough.

A plot of the effluent metal analyses with respect to time at each voidage indicates the extraction column retention time available to yield the desired effluent quality or breakthrough point. In a continuous operation, an increment of loaded resin is transferred periodically. Thus, knowing the retention time and transfer period, the increment of loaded resin for transfer can be calculated. The resin increment is kept around 10 vol % of the bed volume or less, so as not to unduly disturb the bed flow pattern. Resin transfer volume for the 51-mm unit



(1) water inlet

(2) washed resin outlet

(3) polyethylene section

(4) 50.8 mm (2 in.) x 1.22 m (48 in.) pipe

(5) drain line

(6) 19.05 mm (3/4 in.) PVC ball valve

(7) eluant solution inlet

(8) 50.8 mm (2 in.) and 101.6 mm (4 in.) coupling

(9) 50.8 mm (2 in.) x 2.08 m (82 in.) pipe

(10) 101.6 mm (4 in.) x 50.8 mm (2 in.) reducer

(11) photo-electric cell

(12) 101.6 mm (4 in.) x 0.965 m (38 in.) pipe

(13) eluate solution outlet

(14) loaded resin inlet

(15) 50.8 mm (2 in.) and 101.6 mm (4 in.) coupling

Fig. 5 - Wash and elution columns

is approximately 170 mL wet settled resin (w.s.r.) every 10 to 20 min and this volume is transferred in about 8 s. Retention times in elution and washing are developed during the continuous runs.

CONTINUOUS OPERATION

PRE-LOADING OF RESIN

Continuous operation requires pre-loading of resin in the extraction column before automatic operation can be started.

As soon as the correct amount of resin has been placed in the column to obtain the desired voidage, the pre-loading phase may start by passing the feed solution through the bed, keeping the expanded height constant. Voidage will consequently also remain constant. Duration of the pre-loading period is given by the time required to reach the breakthrough point as determined during the static run at a set voidage. Ordinarily, duration of the pre-loading period is decreased by one hour to be certain of reaching the desired effluent quality when automatic operation begins. When the pre-loading phase is completed, automatic operation is begun. If automatic operation does not take place immediately after pre-loading, washing the resin bed with water to remove feed solution that still is in contact with the resin is recommended.

AUTOMATIC OPERATION

Starting up requires manual adjustment of the amount of resin placed in the different columns of the circuit as follows:

i The resin in the extraction column is expanded with water until a steady fluidized bed is achieved at the predetermined height, h.

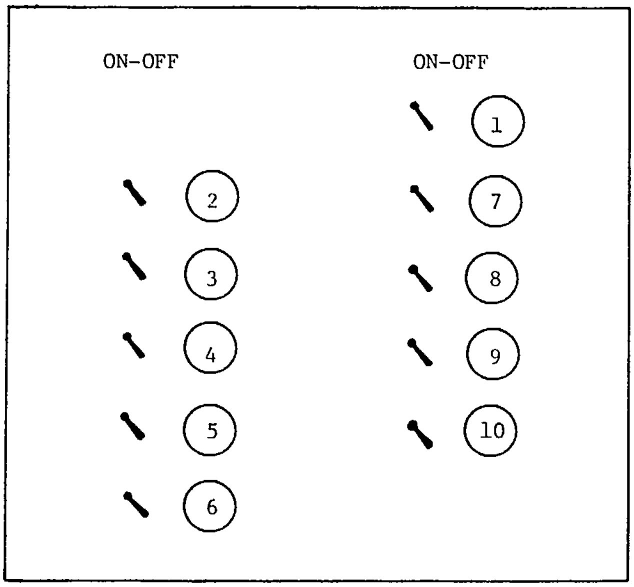
ii The master panel main switch is turned on (Fig. 6).

iii The resin in the wash and elution columns is expanded slightly according to the eluant and water flows used to give an expanded resin height in the regeneration column which is slightly below the photoelectric cell level. This is done by turning the switch on for the peristaltic pumps for eluant and water solutions.

The ratio of eluant to water depends on the strength of eluant desired. The solution is prepared at a higher strength initially to compensate for the dilution factor from wash water.

iv The loaded resin transfer water pump is started by turning the switch on. Water is used as transfer solution at the start but as soon as enough eluate product is available, that solution is then used.

v The compressed air valve is opened to operate automatic ball valves.



1 Master panel main switch
2 Timer-automatic ball valve
3 Photo-electric-relay
4 Photo-cell light
5 Eluant solution pump & relay box
6 Centrifugal pump (vacuum system)
7 Agitator in eluate tank
8 Water wash pump
9 Feed solution pump
10 Transfer solution pump

Fig. 6 - Master panel control board

vi The vacuum pump is turned on. At this point the resin should be in balance in each column of the circuit, i.e., the desired expanded heights in the extraction and regeneration columns should have been reached; continuous automatic operations may start.

vii Water flow through the resin bed of the extraction column is stopped and replaced by feed solution flow.

viii The photoelectric cell and light source switches are turned on.

ix The timer for the resin increment is turned on, ordinarily at a frequency of 10 min between two loaded resin discharges. The resin increment is set by the delay on the timer in seconds.

x Fine adjustment may be needed to obtain the desired resin increment by decreasing or increasing the delay on the timer which controls the opening duration of the automatic ball valve.

The first step in the procedure may be omitted if the continuous run takes place immediately after the pre-loading period ends.

REFERENCES

1. Slater, M.J. "A review of counter-current contactors for liquids and particulate solids"; Br Chem Eng; 14(1):41-46; 1969.

2. Slater, M.J. "Continuous counter-current ion exchange in the mining industry"; Can Ind Metall Bull; 93-98; February 1974.

3. Slater, M.J. "Continuous ion exchange in fluidized beds"; Can J Chem Eng; 52:43-51; February 1974.

4. Slater, M.J. "Assessment of fluidized bed ion exchange equipment"; J Appl Chem Biotechnol; 25:367-378; 1975.

5. Slater, M.J. and Prud'homme, P. "Continuous ion exchange in fluidized beds"; Mines Branch, [since renamed Canada Centre for Mineral and Energy Technology (CANMET)], Energy, Mines and Resources Canada; Tech Bull TB 158; 1972.

6. Lucas, B.H. "Separation processes by single stage deep fluidized bed continuous ion exchange"; CANMET, Energy, Mines and Resources Canada; MRP/MSL 78-159 (OP&J); 1978.

APPENDIX A - LIST OF PARTS

EXTRACTION COLUMN

Kimax beaded glass tube - 51 mm diam x 244 m long

Plexiglass base - 51 mm diam with two 12.7 mm diam holes for feed intake and loaded resin withdrawal

Stainless steel top plate - 51 mm diam with two 12.7 mm diam holes for raffinate overflow and incoming washed resin

Polyethylene funnel as incoming washed resin receiver

12.7-mm diam non-return valve on feed inlet line

Standard varidrive model 240 A peristaltic pump for pumping feed solution into extraction column base

12.7-mm diam Jamesbury automatic ball valve for loaded resin withdrawal from extraction column base

Timer, (0-120 min) (0-60 s) range to control opening and closing of the loaded resin automatic ball valve

1/4 hp finger pump to recirculate eluate product and to pump loaded resin into the regeneration column

Compressed air source comprising air regulator and air gauge for the automatic ball valve

ELUTION COLUMN

Kimax beaded glass tube - 51 mm diam x 208 m long

Kimax beaded glass tube - 102 mm diam x 610 m long

Kimax beaded glass reducer T tube - 102 mm diam x 356 mm long x 51 mm diam side arm

Kimax beaded glass reducer tube - 102 mm to 51 mm diam

Polyethylene base - 51 mm diam with a 19.1-mm vertical hole in centre and a 12.7-mm diam side-hole for eluant intake

Stainless steel side plate - 51 mm diam with a 12.7 mm diam hole in centre as eluate product outlet

12.7 mm diam PVC ball valve on eluant intake line

19.1 mm diam PVC ball valve at base of elution column

Photoelectric cell and a light source which controls resin level in solution column

Dual time-delay photoelectric relay which controls vacuum action

Relay box coupled with photoelectric relay to control eluant solution pump cut-off when pulling washed resin into the extractor

Micro-M model 100 peristaltic pump for feeding eluant solution

WASH COLUMN

Kimax beaded glass tube - 51 mm diam x 1.22 mm long

Polyethylene top - 51 mm diam with a 19.1-mm diam hole

Polyethylene base, 51 mm diam with a 12.7-mm diam vertical hole in centre as wash water inlet and a 12.7-mm diam side-hole as washed resin outlet

Micro-M model 100 peristaltic pump for feeding water wash

12.7-mm diam PVC ball valve on drain line

VACUUM SYSTEM AND HOPPER DEVICE

Three-way solenoid valve

Water injectors

Kimax beaded glass reducer - 152 mm to 51 mm diam as hopper

12.7 mm diam Jamesbury automatic ball valve controlled by the photoelectric cell for discharging washed resin from the hopper into top of extraction column

Compressed air source comprising air regulator and air gauge for the automatic ball valve

Plexiglass top plate - 152 mm diam as hopper cover

Stainless steel plate - 51 mm diam as hopper base

1/2 hp Eastern centrifugal pump for recirculating water through water injectors to create vacuum

Master panel control board which controls all the operation (Fig. 6)