Mines Branch Information Circular IC 259

CONSTRUCTION AND OPERATION OF AN EXPERIMENTAL SIEVE-PLATE PULSE COLUMN FOR SOLVENT EXTRACTION OF METALS

by

G.M. Ritcey*, B.H. Lucas*, K.T. Price** and R.L. Chagnon**

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ABSTRACT

One method used in the separation and recovery of metals during hydrometallurgical processing is liquid-liquid extraction, commonly referred to as solvent extraction. Various types of equipment have been used to effect the mass transfer of the metal across the organic-aqueous boundary. For systems requiring up to four theoretical stages, mixer-settlers are adequate; but, for systems where the partition coefficients are small, therefore necessitating many stages of extraction, a device such as an agitated column is desirable. This report describes one such apparatus, the Sieve-Plate Pulse Column, which has been used successfully in plants for solvent extraction processing. The construction of this multi-stage extraction device is described, and the report provides a list of all the necessary accessories for assembling an experimental 2-inch-diameter, 40-foot-high glass column. A 2-inch-diameter column has been found to be the minimum size from which useful data for plant-scale column design can be obtained.

*Research Scientists and **Technicians, Hydrometallurgy Section, Extraction Metallurgy Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

Direction des mines

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CONSTRUCTION ET FONCTIONNEMENT D'UNE COLONNE PULSÉE À PLAQUES PERFORÉES EXPÉRIMENTALE, DESTINÉE À L'EXTRACTION DES MÉTAUX PAR SOLVANT

par

G.M. Ritcey*, B.H. Lucas*, K.T. Price** et R.L. Chagnon**

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RÉSUMÉ

Une des méthodes utilisées pour la séparation et la récupération des métaux dans le traitement métallurgique par voie humide est l'extraction liquide-liquide, mieux connue sous le nom d'extraction par solvant. Plusieurs types d'appareils ont été employés pour réaliser le transfert en masse du métal à travers l'interface matière organique-eau. Pour des mélanges n'exigeant théoriquement pas plus de quatre étages, des mélangeurs-décanteurs suffisent; mais, pour des mélanges présentant de faibles coefficients de séparation et qui nécessitent, de ce fait, de nombreux étages d'extraction, un dispositif du genre colonne à agitation s'impose. Le présent rapport décrit un appareil de ce type, la colonne pulsée à plaques perforées, qui a été utilisée avec succès dans des usines de traitement employant le procédé d'extraction par solvant. Les auteurs décrivent la construction de ce dispositif d'extraction à étages multiples, et le rapport contient la liste de tous les éléments nécessaires pour assembler une colonne de verre expérimentale de 2 pouces de diamètre et haute de 40 pieds. Les auteurs ont découvert qu'une telle colonne devait avoir au moins 2 pouces de diamètre, si l'on veut en tirer des données utiles pour l'étude d'une colonne à l'échelle industrielle.

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*Chercheurs scientifiques et **techniciens, Section de l'hydrométallurgie, Division de la métallurgie extractive, Direction des mines, ministère de l'Énergie, des Mines et des Ressources, Ottawa, Canada.

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INTRODUCTION

In an organic aqueous two-phase solvent extraction process, the mass transfer of the desired metal from one phase to the other is accomplished by the use of various types of equipment in which the aqueous stream is usually flowing countercurrently to the organic stream. Because of the simplicity and flexibility of the solvent extraction process, a wide choice of operating conditions is often available. The design of the process and the proper selection of equipment for scaling up from the bench to the plant then become important aspects in developing favourable economics.

The choice of a contactor suitable for any particular solvent extraction system will be dictated by the following: First, the special physical-chemical requirements, such as contact time, settling characteristics, floor area, or head room, must be satisfied. Next, the number of contact stages and the desired throughput must be considered, as well as the increasing cost of maintenance as the contactor becomes more complex. From these considerations the contactor having the lowest cost factors and highest suitability for the specific extraction operation can be ascertained.

The most common equipment for solvent extraction work can be classified as either stagewise or differential contactors. In a stagewise contactor, such as a mixer-settler, the two immiscible phases are mixed and then allowed to separate or disengage, because of their difference in density, in a separate settling chamber. Differential contactors, such as columns, provide continuous contacting and therefore many mixing, settling or coalescing stages. Although mixer-settlers are still very popular for systems requiring up to four stages and for high throughput rates, they occupy a large area and require a high solvent inventory for a given capacity.

In column extractors, differences in the solution densities are utilized for countercurrent vertical flow through a column. Generally the aqueous input is located at the top of the column and the organic input is located near the bottom. Essentially, columns can be divided into two classes: non-agitated, and agitated. In the non-agitated class are, for example, packed columns containing discs or rings, spray columns, or sieve-plate columns; none of these types has proved to be very successful in industry, because of relatively low efficiencies and throughputs.

Agitated columns have had a greater application in industrial processing. Among these is the Schiebel column(1), which consists of a number of mixing zones in which the phases are mixed by an impeller mounted on a central rotating shaft; the mixing zones are separated by settling zones which are either baffled or contain packing. The Oldshue-Rushton(2) column contains baffles spaced down the length of the column, and agitation is accomplished by a centrally driven shaft on which paddle mixers are mounted. This latter extractor has been improved several times, resulting in units such as the Rotating-Disc Contactor(9) and also the Mixco Column manufactured by Greey Mixing. All these columns have certain disadvantages in solvent extraction for metal recovery, such as a low tolerance in the amount of suspended solids present in the feed material, which results in an emulsion forming in the column. Also, a 5 to 10% spread in rotation speed can often be the difference between insufficient agitation and emulsification.

Another type of agitated column is the pulsed column. In this type, either the solution is pulsed, as in the pulsed sieve-plate column(3,4), or the plates are pulsed through the solution in a reciprocating motion, as in the Karr reciprocating plate column(5). In the reciprocating plate columns, diameters of up to 20 inches have been used industrially(6), while in the sieve-plate pulse column, diameters up to 34 inches have been used in the nuclear energy field(7). Pulsation is effected, in the reciprocating plate column, by an eccentric cam mounted on the top of the column. In the sieve-plate pulse column, the energy generally has been supplied by a bellows or diaphragm pump. Recently, Baird has reported on the use of air pulsation(8), but this has not been developed to a commercial scale.

The preceding description of other extraction equipment, with reference to column extractors in general, has been kept intentionally brief, and the reader can refer to other sources for more detailed descriptions[9-11]. The emphasis at the Mines Branch in the past three years has been on solutions containing several metals where the separation factors between metals is relatively small. Consequently, a contactor was required that was capable of providing many theoretical stages of extraction in order to attain separation between metals having similar extraction coefficients. The pulsed sieve-plate column was chosen because of its successful use in the nuclear industry[12] as well as in systems requiring many theoretical stages[13-17]. Because of the gentle pulsating action to achieve mass transfer, the pulsed column shows promise for the extraction of metals from leach slurries[18]. Also, a glass experimental column is easy to construct and operate. Because of the excessive wall effects, columns of diameter less than 2 inches produce experimental data that are unsuitable for scale-up calculations. A description is given for the construction and operation of a 2-inch-diameter, 40-foot-high, glass sieve-plate pulse column, shown diagramatically in Fig. 1.

PULSE COLUMN CONSTRUCTION

The overall length of the column described in this report is approximately 40 feet, standing vertically on a 3-foot-high support-stand constructed of standard black angle-iron (2" x 1/8"), which may be adjusted up or down 6 inches (Fig. 2). With this type of stand, the operator, after having clamped the upper sections

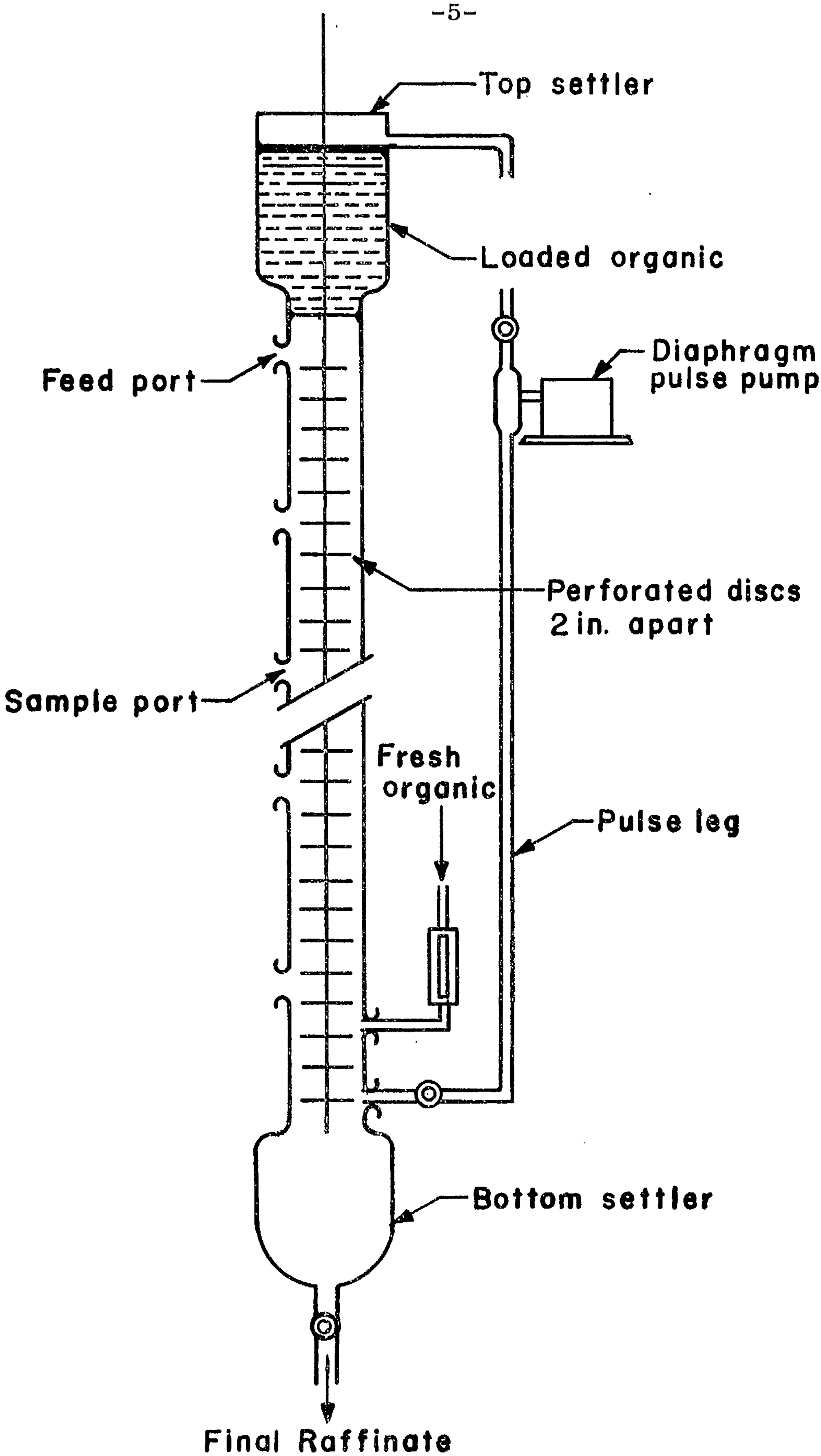


Fig. I. The sieve-plate pulse column.

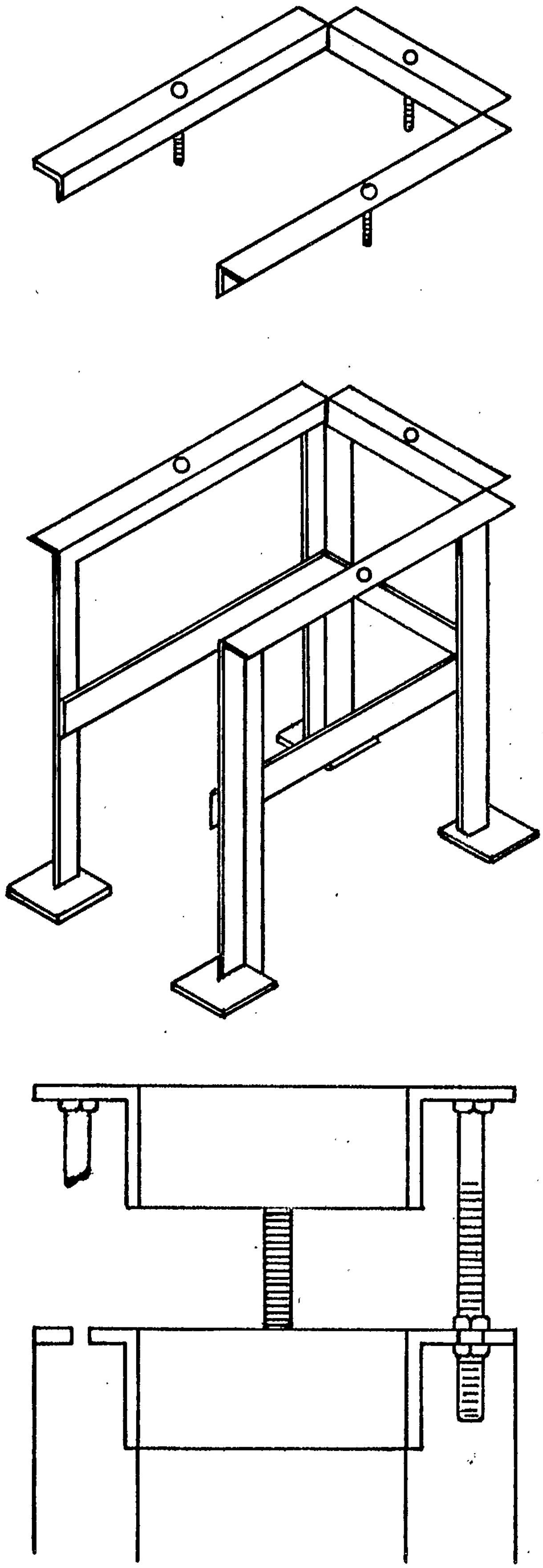


Fig. 2. Column support stand.

securely, can remove the bottom settler for cleaning and maintenance. The column is made of tempered 2-inch glass piping of various lengths. Two-inch glass T's are used for sample and feed inlet ports, while glass reducers are used for top and bottom settling chambers. The detailed list of glass pipe and type 316 stainless steel fittings is shown as an Appendix to this report. There are five sample and/or feed inlet ports located up the column. These ports enable the column to be operated with a variety of effective heights in order to evaluate the efficiency of the process for scale-up purposes.

The support pipe (Fig. 3) to which the glass column will be fixed is made of standard black iron, $1\frac{1}{4}$ to $1\frac{1}{2}$ inches in diameter. The pipe is connected to a pipe flange which is bolted to the floor. Clamps are welded at each floor level (13-foot intervals) to hold the pipe in place. Holes are drilled 2 feet apart through the pipe, to accommodate 3-prong clamps which hold the glass column to the vertical pipe. Circular clamps of 4-inch and 6-inch diameter are used to support the top and bottom settlers.

The support stand is placed beside the support pipe and the glass column is assembled, beginning with the bottom settler and working upwards. The glass pipe is connected by means of flange sets made for the purpose by the manufacturer. The flanges are sealed with teflon "T" gaskets. As excessive tension may cause the 2-inch-diameter glass lengths to crack, care should be taken,

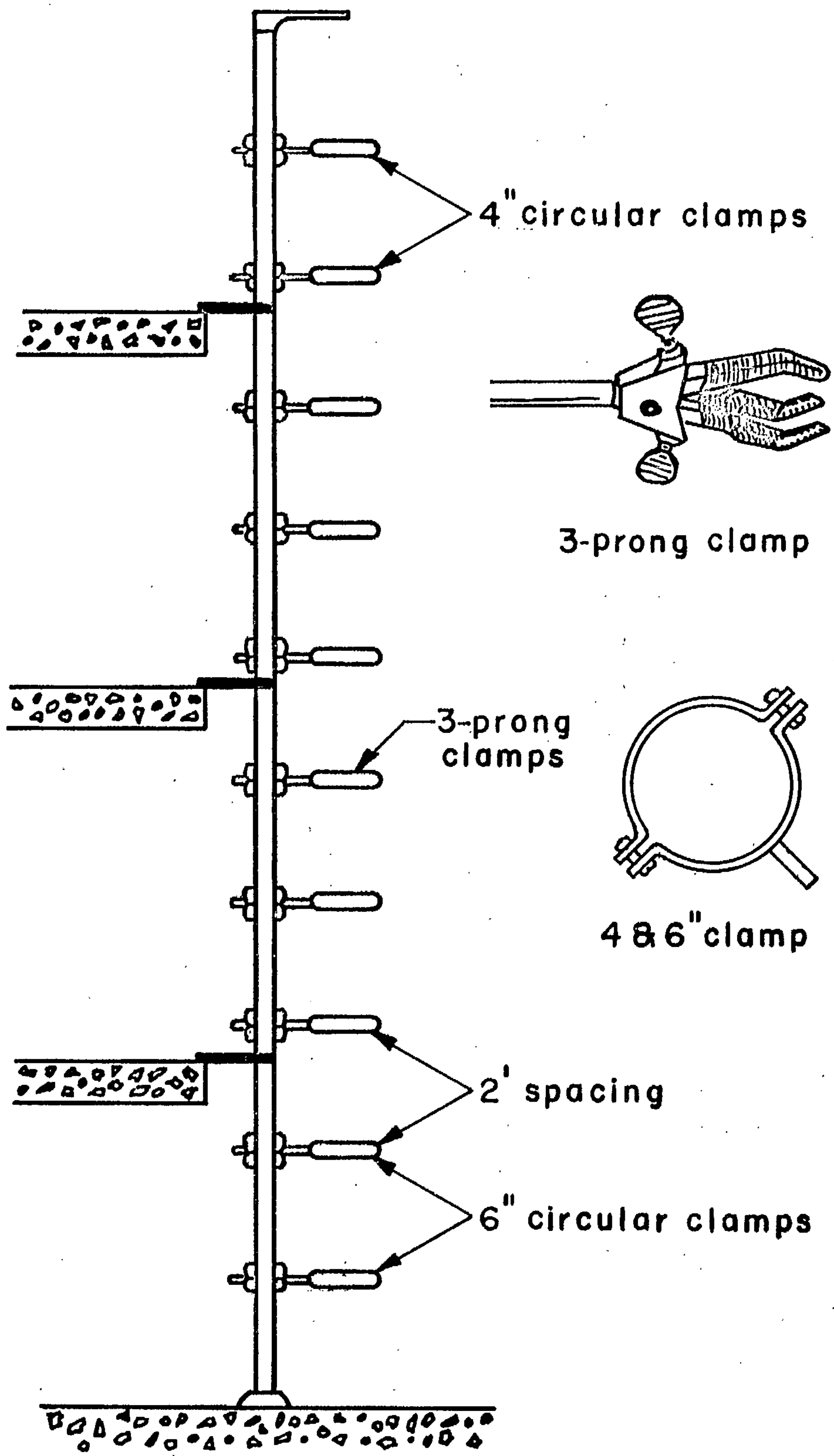
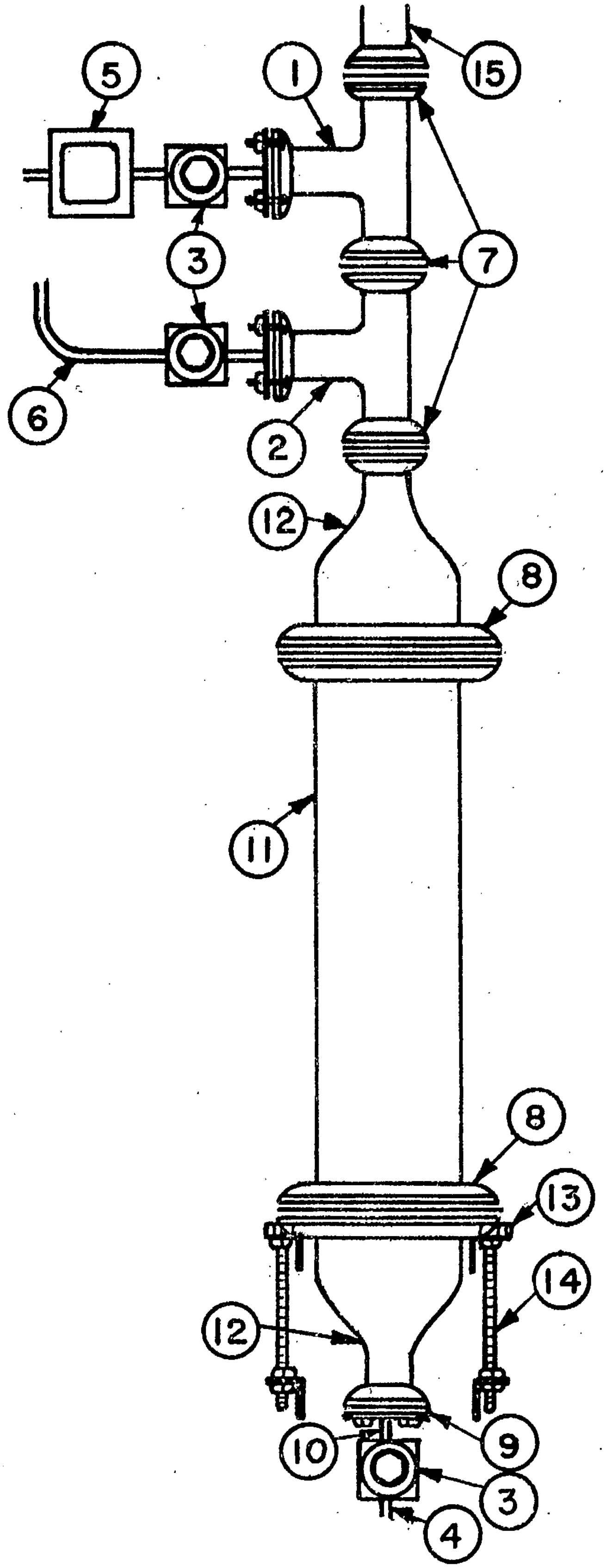


Fig. 3. The column support pipe.

with the aid of a level, to keep the column perpendicular. That is, the bottom settlers are placed together, reducing to the 2-inch-diameter size as shown in Fig. 4. Next, the 2-inch T (No. 2) for the pulse leg, and then the 2-inch T (No. 1) for the organic inlet, are affixed to the bottom settler (Fig. 4). The desired column lengths and sample T's or crosses are then fixed in place (Fig. 5), followed finally by the top settler as shown in Fig. 6.

The support rod which runs through the centre of the column is used to carry the sieve-plates (Fig. 5). It is constructed of ¼-inch-diameter, type 316 stainless steel rod cut to 6-foot lengths and connected with 2-inch-long connectors made from 7/16-inch-diameter, type 316 stainless steel rod, and threaded at both ends. Additional lengths of rod without the spacers and sieve-plates may be extended up through the top settler and suspended from a bracket attached to the column support pipe. This will hold the rod and sieve plates in place. The sieve plates are spaced 2 inches apart, using spacers made from 1/8-inch-diameter schedule 40, type 316 stainless steel pipe. A plate spacing of 2 inches was chosen by this laboratory because most industrial-size pulsed columns employ such a plate-spacing[9], and therefore scale-up data would be meaningful.

Perforated (sieve) plates are used in the column for the formation of numerous fine bubbles to provide a large surface area across which to transfer the desired metals. The plates may



1 Organic feed port
2 Pulse entrance port
3 Gate valves
4 Raffinate exit port
5 One-way valve
6 Pulse leg
7 2" flanges & gaskets
8 6" flanges & gaskets
9 2" flange & gasket
10 Single outlet, s-s, blank-off plate.
11 6"X2' pipe
12 6"X2" reducer
13 1" spacer
14 Column levelling support stand
15 2" glass pipe

Fig. 4. Bottom settler.

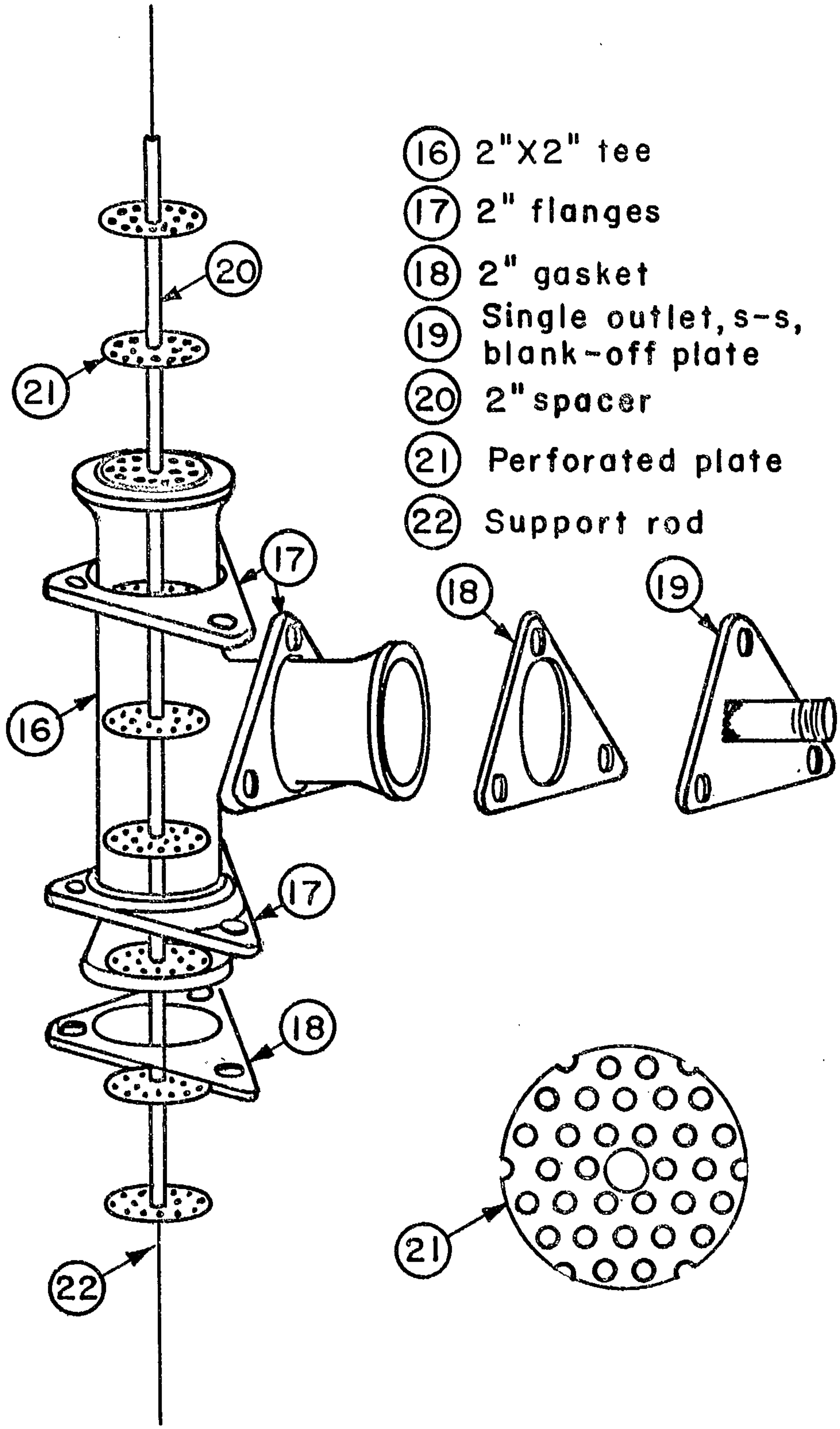


Fig. 5. Feed and sample ports.

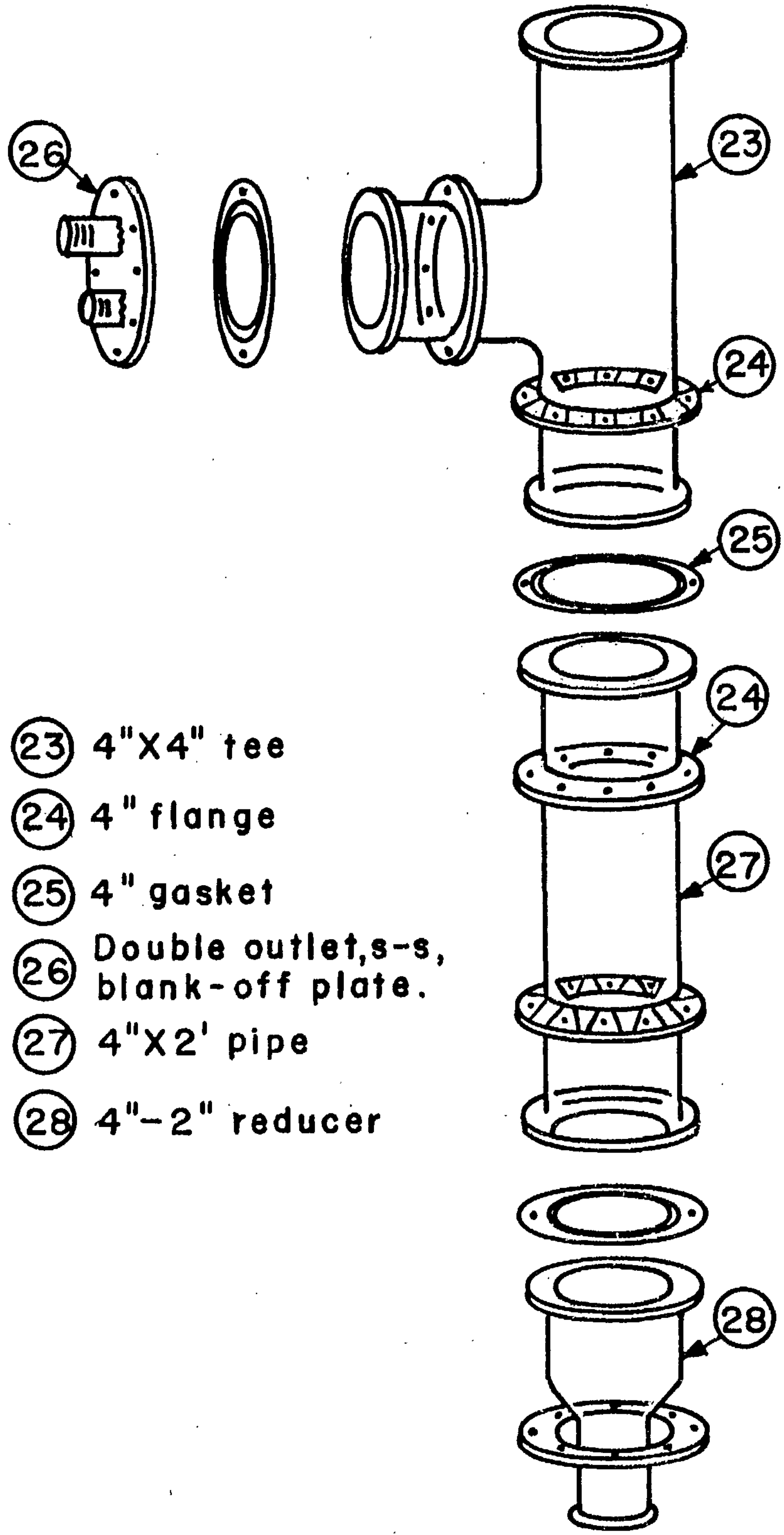


Fig. 6. Top settler.

be made from stainless steel or of teflon sheeting (Fig. 5). The hole diameter, angle of holes, and free area are optional. In experimental work at the Mines Branch we generally start with stainless steel plates containing 3/16-inch-diameter holes with about 30% open area. Sufficient plates for a 40-foot column are obtained by cutting circles from a sheet of perforated stainless steel plate ordered as: 36-inch x 36-inch x 18-gauge, punched with 3/16-inch round holes on 5/16-inch staggered centres, type 316 stainless steel. The circles are cut to a diameter of 1.95 inches in order to provide easy clearance between the plate and the 2-inch inside diameter of the glass pipe, buffed to remove any sharp edges, and centre-punched to permit the support rod to be inserted. If teflon plates are desired, circles may be cut from a sheet of teflon size: 24 inch x 48 inch x 1/8 inch. The bottom sieve-plate should be placed about 4 inches above the organic feed port (Fig. 4) which is near the bottom of the pulse column. The top plate should be about 4 inches below the top feed port (Fig. 1).

The tees (T's) (Fig. 5) which make up the sample or feed ports require blank-off plates of type 316 stainless steel (1/8 inch thick), with a 3/8-inch-diameter hole cut in the centre to accommodate a 3/8-inch stainless steel nipple which is welded to the blank-off plate. 3/8-inch gate valves, type 316 stainless, are threaded to these nipples. If 2- x 2-inch straight crosses are used instead of tees, blank-off stainless steel plates, minus the nipples, will be required to close off the other side of the cross, or this other side may be used to insert a probe for pH or emf measurements, etc.

A 3/8-inch stainless steel check-valve should be installed on the organic feed inlet port (Fig. 4) to prevent the aqueous feed from flowing back through the organic line. A 3/8-inch gate valve may also be installed here, as in Fig. 4, for safety reasons. The pulse inlet valve and the raffinate discharge valve at the bottom of the column should be of ½-inch size. The double-outlet stainless-steel blank-off plate on the top settler (Fig. 6) has two ½-inch nipples welded to it. The bottom nipple is to accommodate the organic overflow line, while the top nipple is for emergency use should the column throughput be exceeded. Any overflow from here is directed into a suitable container through a tygon hose.

The pulse pump is a Denver 3/4-inch laboratory suction-pressure diaphragm pump with the check-valve balls removed, complete with a ¼-h.p. single-phase, 60-cycle, 110-volt motor and a variable speed adjustment. The pump head and check valves are all of type 316 stainless steel. Any type of pump that will give a good pulse may be used. Metering pumps, or standard pumps with the flow measured through flow-meters, are also required for solvent feed, aqueous feed, and raffinate discharge.

A parts list is given in the Appendix.

OPERATION

The test work on the experimental 2-inch-diameter pulsed column should be planned so as to cover a range of flow rates, up to the flooding point of the column. The flow ratios are generally fixed and previously determined by bench shake-outs, and dictated by the saturation capability of the organic and the concentration of the extractable metal in the feed. Also, a range of pulse conditions should be tested, as well as plate-hole geometry, wetting characteristics of the plate material, and variation of the continuous phase in the column.

Prior to start-up, the pulse leg is filled with the organic solution, with the pulse leg inlet-valve shut off. The column is then filled with the desired solution for the continuous phase. For example, in an aqueous continuous system, the aqueous is pumped in, followed by the addition of sufficient organic to bring the organic-aqueous interface in the region of the lower part of the top settling chamber. The organic and aqueous flows are then metered in at the desired flow ratios and rates, the pulse leg valve is opened, and the pulse pump is turned on. The pulse amplitude and frequency are then adjusted on the pulse pump as determined by visual observation of the bubble formation and coalescence at the plates. The amplitude of the pulse ranges from 1 to 2 inches, and the frequency between 20 and 60 pulses per minute. In our experience, the total flow capacity depends upon the system itself, viscosity, specific gravity difference between the organic and aqueous solutions, amount of solids present, hole diameter, and type of sieve plates. The total throughput has ranged between 1,000 and 2,000 ml per minute in this experimental 2-inch-diameter sieve-plate pulse column. It must be stressed that the conditions to produce bubbles of a specific size, as well as the proper coalescence in one system, may not be the same conditions necessary for another system. In Table 1 are listed some problems normally encountered in pulsed-column operation, together with some solutions to the problems.

CONCLUSION

This report has described the construction of a 40-foot-high, 2-inch-diameter, glass pulsed sieve-plate column, and also has provided a list of all the required parts for the construction. The column is of sufficient diameter and height to provide the necessary pilot-plant data for scale-up to plant operation for liquid-liquid or slurry extraction systems.

TABLE 1

Some Problems Encountered in Pulsed-Column Operation, and Possible Remedies

Observation	Cause	Remedy
Inversion of phases	1. Flooding	1. Decrease flows or increase hole diameter in plates
	2. High viscosity	2. Decrease flows or heat feed solutions
Emulsion condition	1. Very fine bubbles	1. Decrease pulse frequency and/or amplitude
	2. Wetting characteristics of plates	2. Change material of sieve plates
	3. Excessive backmixing	3. Trim pulse leg valve
	4. Flooding	4. Decrease flows
Poor coalescence	1. Very fine bubbles	1. Decrease pulse frequency and/or amplitude or perhaps an additive required
	2. Wetting characteristics of plates	2. Change material of sieve plates
Poor extraction	1. Column too short	1. Increase column height
	2. Wrong bubble size	2. Change pulse conditions or decrease hole diameter in plates
	3. Poor coalescence	3. Change pulse frequency and/or amplitude
	4. Inefficient agitation	4. Increase pulse frequency and/or amplitude
High organic losses in aqueous	1. Inadequate settler	1. Increase column height
	2. Excessive pulsing	2. Decrease pulse frequency and/or amplitude
	3. Partial emulsion	3. Chemical additive to improve phase disengagement
	4. Excessive flows	4. Decrease flows
	5. Organic concentration too high	5. Decrease concentration
High aqueous entrainment in organic	1. Inadequate settler	1. Increase settler area
	2. Excessive flows	2. Decrease flows

REFERENCES

1. Shiebel, E.G., "Performance of an Internally Baffled Multistage Extraction Column", A.I.Ch.E. Journal 2, No. 1, pp. 74-78 (1956).
2. Oldshue, J.Y., and Rushton, J.H., "Continuous Extraction in a Multistage Mixer Column", Chemical Engineering Progress 48, No. 6, pp. 297-306 (June 1952).
3. Richardson, G.C., and Platt, A.M., "The Design and Operation of Industrial Scale Pulse Columns for Purex Service", Technology, Engineering and Safety, Progress in Nuclear Energy, Pergamon Press Pub., New York, 1961, Series IV, 4, pp. 279-307.
4. Sege, G., and Woodfield, F.W., "Pulse Column Variables", Chemical Engineering Progress 50, No. 8, pp. 396-402 (Aug. 1954).
5. Karr, A.E., "Performance of a Reciprocating-Plate Extraction Column", A.I.Ch.E. Journal 5, No. 4, pp. 446-452 (Dec. 1959).
6. Prochazka, J., Landau, J., Souhrada, F., and Heyberger, A., "Reciprocating-Plate Extraction Column", Inst. of Chemical Fundamentals, Czechoslovak Academy of Sciences, Prague. Presented at the CHISA Congress, 1969.
7. Mar, B.W., and Babb, A.L., "Longitudinal Mixing in a Pulsed Sieve-Plate Extraction Column", Ind. Eng. Chem. 51, pp. 1011-1014 (1959).

8. Baird, M.H.I., Gloyne, A.R., and Meghani, A.N., "Solvent Extraction in an Air-Pulsed Packed Column", Canadian Journal of Chem. Eng. 46, pp. 249-252 (1968).

9. Treybal, R.E., "Liquid Extraction", McGraw-Hill Publ. Co., New York (1963).

10. Hanson, C., "Solvent Extraction", Chem. Eng. 75, No. 18, pp. 76-98 (1968).

11. Hanson, C., "Liquid-Liquid Extraction Methods and Equipment", Brit. Chem. Eng., Equipment Supplement, pp. 49-57 (Nov. 1968).

12. Burger, J.C., and Jardine, J.M., "Canadian Refining Practice in the Production of Uranium Trioxide by Solvent Extraction with Tributyl Phosphate", Proceedings of 2nd United Nations Int. Conf. on Peaceful Uses of Atomic Energy, Geneva, 4, 3-9 (1958).

13. Thunaes, A., and Colborne, G.F., "Pulsed Columns for Solvent Extraction from Aqueous Solution and Slurries"; paper presented at Ninth Commonwealth Mining and Metallurgical Congress, London, May 1969.

14. Ashbrook, A.W., Ritcey, G.M., and Joe, E.G., "Mixed Extractant and Process for Separating Copper from Cobalt and Nickel in Acid Medium", Journal of Metals, AIME, 18, No. 1, pp. 18-21 (1966); U.S. Patent 3,455,680 (1969).

15. Ritcey, G.M., and Ashbrook, A.W., "Separation of Cobalt and Nickel by Liquid-Liquid Extraction from Acid Solution", U.S. Patent 3,399,055 (Aug. 1968).

16. Ritcey, G.M., and Ashbrook, A.W., "Separation of Nickel and Cobalt in Ammoniacal Solutions by Liquid-Liquid Extraction", Inst. of Mining and Metallurgy, Trans. C, 78, pp. 57-63 (1969); U.S. Patent 3,438,768 (1969).

17. Ritcey, G.M., and Conn, K., "Liquid-Liquid Separation of Zirconium and Hafnium", Report T 67-7, Research and Development Division, Eldorado Nuclear Limited, Ottawa (1967).

18. Ritcey, G.M., Joe, E.G., and Ashbrook, A.W., "Solvent-in-pulp Extraction of Uranium from Acid Leach Slurries", Transactions of SME/AIME, 238, pp. 330-334 (1967).

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GMR:KTP:BHL:RLC:(PES)dmp

APPENDIX - LIST OF PARTS

Glass Pipe, and Accessories

Glass pipe for the construction of the pulse column described may be obtained from such suppliers as the following:

(1) EIVS (Pegasus, Agincourt, Ontario)

(2) Kimax (Montreal, Quebec)

(3) Quickfit (QVF Toronto, Ontario)

Quantity	Item	Part No.
7	2-inch straight "T"	16
1	4-inch straight "T"	23
1	4-inch x 2-inch reducer	28
2	6-inch x 2-inch reducer	12
1	4-inch x 24-inch pipe	27
1	6-inch x 24-inch pipe	11
1	2-inch x 84-inch pipe	15
3	2-inch x 72-inch pipe	15
1	2-inch x 60-inch pipe	15
1	2-inch x 24-inch pipe	15
2	Teflon interface gaskets, 6 inch	8
3	Teflon interface gaskets, 4 inch	25
24	Teflon interface gaskets, 2 inch	18
2	Flange sets style #2, 6 inch	8
3	Flange sets style #1, 4 inch	24
20	Flange sets style #1, 2 inch	17

cont'd.

Quantity	Item	Part No.
1	4-inch double-outlet SS blank-off plate	26
8	2-inch single-outlet SS blank-off plate	19
2	6-inch circular clamps	Fig. 3
2	4-inch circular clamps	Fig. 3
17	3-prong clamps	Fig. 3
1	Wood spacer 1 inch thick x 12 inch diameter	13

Stainless Steel Fittings

6	3/8" gate valves
1	3/8" check valve
2	½" gate valves
12	3/8" x 2" nipples
6	½" x 2" nipples

Tygon Tubing

The type of tubing required for the pulse column can be optional but should be of Tygon whenever in contact with any organics. The suggested sizes are as follows:

50 feet ½" I.D. x 3/32" wall

100 feet 3/8" I.D. x 1/16" wall

50 feet vacuum tubing, Tygon ¾" I.D. x 3/8" wall, used on the pulse pump.

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