256. *A Small Laboratory Liquid–Liquid Extraction Apparatus.*

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A convenient apparatus is described suitable for routine liquid–liquid extraction on the laboratory scale. It is simple to construct and operate and offers distinct advantages over the conventional method of extracting by batch-wise shaking in separatory funnels.

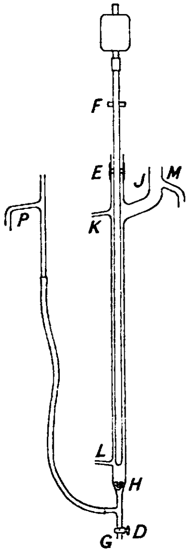
THE extraction of a liquid by an immiscible or comparatively immiscible solvent is normally carried out in the laboratory by the inefficient method of repeated single-stage shakings with fresh lots of solvent; with unfavourable distribution coefficients, this leads to a relatively large bulk of weak extract, and is tedious and time-consuming, particularly where phase separation is difficult. A considerable advantage should be obtained in such operations by use of a counter-current extraction column where a multi-stage separation can be effected in a single pass. Such an apparatus can be operated under conditions which minimise phase-separation difficulties by avoiding extremely fine dispersion.

Most existing types of column are too large and complicated for small-scale application, but one simple type, capable of being scaled down to suitable dimensions, was first described by Jantzen (Dechema Monograph 5, No. 48, p. 114, 1932) and improved by other workers (Ney and Lochte, *Ind. Eng. Chem.*, 1941, **33**, 825; Schutze *et al.*, *Ind. Eng. Chem. Anal.*, 1938, **10**, 675; Shell Development Co., B.P., 615,425/1949; Short and Twigg, *Ind. Eng. Chem.*, 1951, **43**, 2932). In the simple modification now described, the scale has been reduced so as to afford an effective tool for routine laboratory extractions.

The essential feature of the apparatus is a solid cylindrical core rotating inside a coaxial cylindrical column, and the two relatively immiscible liquids pass counter-currently through the annular space. At certain speeds of rotation the liquid motion in the column assumes the form of a stack of horizontal vortex rings, a plainly visible phenomenon investigated mathematically by Taylor (*Phil. Trans.*, 1923, *A*, **223**, 289) and by Lewis (*Proc. Roy. Soc.*, 1927—28, *A*, **117**, 388). The importance of this motion, as applied to extraction, is that the solvent phase traverses a very long path through progressively stronger raffinate as it passes through the column.

EXPERIMENTAL

Apparatus.—The column was made in one piece from a length *EG* of straight glass tubing of fairly uniform bore, as shown in the diagram. Rotors of different diameters were made from straight lengths of glass rod or sealed tubing or of stainless steel, and, when in use, each was mounted in two bearings cut from discs of 3/16″ " Tufnol " sheet. One bearing was clamped to the top of the column at *E* by a rubber sleeve, and the other held in a clamp 4—5″ above, at *F*. The free end of the rotor was connected by rubber sleeves, by way of a short length of steel rod (to give a measure of flexibility in alignment), to the driving shaft of a 1/30 H.P. variable-speed motor. Owing to the absence of a bearing at the bottom of the rotor, whipping of the latter was pronounced at some speeds of rotation, but this did not appear to affect adversely the efficiency or throughput. Rotor speed was conveniently measured and controlled with the help of a stroboscopic disc mounted on the free end of the motor shaft and actuated by the 50-cycle lighting circuit.



In testing the column, liquids were fed by gravity through small needle valves and rotameters, since this required careful control of flow rates; but for routine operations they could probably be supplied direct from graduated tap-funnels. Lighter phase entered the column at *L* and overflowed at *M* where the " Claisen head " formed a calm space for phase separation when the interphase was at the top. Heavier phase entered at *K* and overflowed at *P*, the level of which could be adjusted to control the position of the interphase in the column. A loose coil of fine-gauge " stainless " steel wire was located at *H* and helped to prevent finely-dispersed lighter phase from being carried out with the heavier effluent. The column could be drained through *D*. Column dimensions are given in Table 1.

Column Operation.—It was important that, with small columns of this kind, the column and rotor should be initially clean and free from grease. With a given extraction system and a given annulus width, there was an optimum rotor speed giving the most efficient extraction. This speed occurred in the range where liquid motion in the column assumed the vortex form. The droplet size of the disperse phase was remarkably uniform and decreased with increasing rotor speed. This uniformity of dispersion and the absence of very finely dispersed liquid,

TABLE 1. *Column dimensions.*

Mean I.D. of tube = 0·772 cm.; overall length, *EG* = 14½ in.; effective annulus length, *KL* = 11½ in.

Rotor	A	B	C	D
Rotor material	Glass	S/S	Glass	Glass
Rotor mean O.D. (cm.)	0·570	0·476	0·456	0·438
Annulus mean width (mm.)	1·01	1·48	1·58	1·67
Free space in assembled column (ml.)	10·6	13·2	13·5	14·0

such as is obtained in the normal batch-wise agitation, helped to minimise difficulty in phase separation. The optimum extraction efficiency generally occurred at rotor speeds of 200—300 R.P.M. above the speed at which the vortex rings first developed. The dispersion was then fine enough for good interfacial contact but not so fine as to reduce throughput seriously by hindering phase separation.

With a given extraction system, the efficiency became greater as the annulus was made narrower, but there was a lower limit to the annulus width for the column to function satisfactorily. This limiting width was usually greater with systems having higher interfacial tension. With an annulus narrower than the limiting width there was a tendency for the disperse phase to coalesce into " slugs " which produced poor interfacial contact and restricted throughput, especially if the apparatus was not perfectly clean. By having a range of readily interchangeable rotors of different diameters it was an easy matter to select one suitable for any of the test systems used. Under standard test conditions, repeatable results were obtained, and Table 2 lists a number of results obtained under test conditions in which the solute concentrations ranged from 3 to 5%. In addition to the test runs, successful column operation was obtained in transferring solutes between water and *iso*propyl ether, decane, and amyl alcohol severally. Column performance was assessed in terms of H.E.T.S., or height equivalent to one theoretical or perfect stage, which may be defined as follows : where the degree of extraction attained in a counter-current extraction column of height H is equivalent to that which would be attained in a batch counter-current series of n perfect stages, the H.E.T.S. is H/n.

An interesting factor in Table 2 is the percentage of total solute transferred in a single passage through the apparatus. This is compared with the percentage which would be extracted in a single equilibrium stage with the same phase ratio. In the test runs the degree of stripping was a secondary condition to the determination of H.E.T.S., and with one exception all test runs were made at approximately the minimum theoretical solvent/feed ratio. The exception was in extracting piperidine from water by carbon tetrachloride at a solvent/feed weight ratio of 2·5, the theoretical minimum being 1·6. This minimum is defined as the minimum ratio at which the solute could be stripped completely in an apparatus comprising an infinite number of stages. When it is realised that the outgoing extract cannot exceed a concentration in equilibrium with the ingoing feed, the minimum ratio will be seen to be approximately the inverse of the distribution coefficient where the latter is truly constant. In practice, a ratio 1·5—2 times the theoretical minimum would be employed, but this would be much less than that required to strip the same percentage of solute by successive extractions with fresh solvent.

TABLE 2. *Test runs with various systems.*

	Continuous phase		Disperse phase					% of total solute transferred in :	
Rotor	Nature	Flow, ml./hr.	Nature	Flow, ml./hr.	Rotor R.P.M.	No. of perfect stages	H.E.T.S., in.	Column	Single stage at same solvent/feed ratio
A	Aq. *cyclo*hexylamine	120	*cyclo*Hexane	60	960	8·0	1·4	91	46
	Aq. acetic acid	67	COMeBu^i	126	960	18·0	0·64	87	50
	COMeBu^i	162	Aq. acetic acid	90	960	4·6	2·5	78	49
	Aq. piperidine	75	C_6H_6	80	580	5·5	2·1	73	49
	,,	120	CCl_4	190	1460	3·7	3·1	93	61
B	COMeBu^i	162	Aq. acetic acid	90	1460	3·0	3·8	68	49
	Aq. piperidine	100	C_6H_6	100	960	3·4	3·4	58	48
	Aq. *cyclo*hexylamine	180	*cyclo*Hexane	90	960	1·7	6·8	63	46
C	Aq. piperidine	80	C_6H_6	80	1460	4·3	2·7	74	48
	Aq. maleic acid	100	Bu^{n}OH	90	580	5·8	2·0	87	49
	Water	100	Maleic acid in Bu^{n}OH	100	820	8·5	1·4	82	49
D	Aq. maleic acid	80	Bu^{n}OH	70	820	7·2	1·6	91	48

Better extraction efficiency was usually obtained as flow rates approached the limits at which the column flooded. The flows quoted in Table 2 are near the respective limits and so give an indication of the throughputs to be expected from such an apparatus. Throughputs are generally lower for the rotating-core column than for other types of comparable size, but this is not a serious handicap to their use on a laboratory scale.

By adjusting the level of the movable overflow, either liquid can be made the disperse phase, and in general more efficient extraction will be achieved by dispersing that liquid which is being passed at the higher flow.

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