A Laboratory Electrodialyser and Desalter

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Electrodialysers described in the literature are for the most part unsuitable for small-scale laboratory use, and details of their performances are not given. Various electrolytic desalters are available which employ a circulating mercury cathode in conjunction with a collodion anode membrane; although widely used in biochemical laboratories they possess several disadvantages. Desalting of a commercial meat extract in such an apparatus was lengthy and resulted in much frothing and evolution of ammonia, owing to decomposition of some of the constituents; finally, the brown colour of the extract was removed, leaving a colourless solution. When meat extract was subjected to exhaustive electro-desalting in the present apparatus none of these gross changes was apparent.

MATERIALS AND METHODS

Meat extract. A commercial extract of meat was dissolved in water and dialysed against distilled water with chloroform as a preservative. The diffusates, free from protein, were combined and concentrated. The solution contained 0·1386 g. of dry matter/ml., 15·1 mg. of N/ml., and 33% of ash.

Nitrogen. This was measured by the semi-micro-Kjeldahl method.

Dry weight. This was determined by evaporation to dryness at 105° in Pt dishes.

Ash. This was determined by igniting the dried samples at 550° in a muffle furnace.

Apparatus

The dialyser (Fig. 1) consists essentially of three square sections cut from 11 mm. Perspex sheet, each bored out in the middle to form a circular chamber 5 cm. in diameter. Two of the sections are closed at one end with a square sheet of 5 mm. Perspex to form the electrode chambers, and into each of these a 9 mm. thickness of 5 cm. diameter carbon rod is cemented to form a sunken electrode 2 mm. below the surface of the section (Fig. 1). Any dead space is filled with Perspex cement.

Holes are bored at each of the four corners, and the three sections when assembled form a block which is held together by wing nuts running on threaded rods passing through the holes. The electrode chambers have holes bored vertically, top and bottom, into which B7 standard ground-glass joints, or cones, are inserted for connexion to the water supply. Communication to the electrode chamber is by drilled-out slots inclined to the membrane in such a way that the water flowing in strikes the membrane, flows across its surface, and flows out through a similar slot, thus reducing stagnation and polarization at the membrane surface.

The centre chamber has a 7 mm. diameter hole in the top. Additional centre chambers are fabricated in the same manner.

Electrical connexion to the electrodes is by short lengths of thin copper rod passing through the plastic and forced into the carbon.

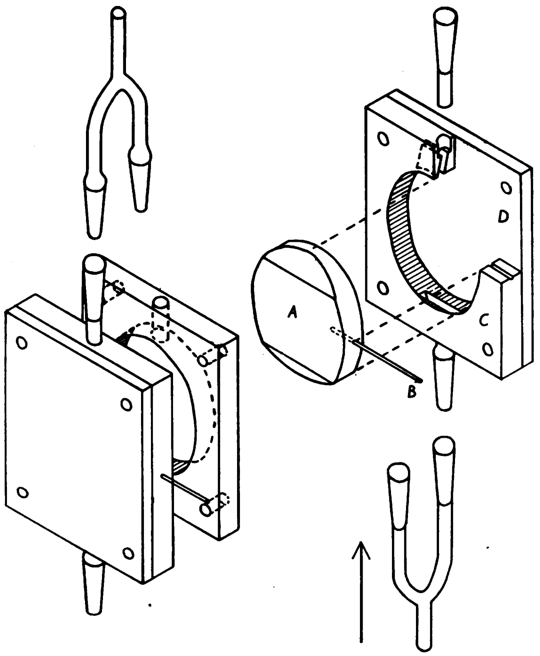

Fig. 1. Isometric view of the electrodialysis cell with one centre compartment in position arranged for desalting. The membranes and the rods and wing nuts are not shown. *A*, carbon electrode 0·9 cm. × 5 cm.; *B*, copper rod; *C*, chamber cut from 1·1 cm. thick Perspex sheet; *D*, Perspex sheet 0·5 cm. cemented to *C*.

Membranes. The membranes are held between the ground faces of the component sections and can be of any suitable material. If there are signs of leakage silicone grease may be applied, or rubber gaskets can be inserted.

Two types of membrane have been tested: ordinary collodion dialysis membrane, and Permaplex ion-exchange membranes (The Permutit Co., London, W. 4). Collodion membranes are suitable for removing smaller molecular species from protein or polysaccharide solutions by electrodialysis. The Permaplex ion-exchange membranes are permeable to inorganic ions and organic ions of size at least up to that of the amino acids, and they are suitable for desalting and electrodialysing non-colloid substances.

Power supply. A direct current of 1000 mA at 20 v was applied at the start of the run, and later, as the resistance of the cell increased, the voltage could be increased to 300 with a corresponding current of 100 mA. The area of the membranes is about 20 cm.^{2}, so that current densities up to 50 mA/cm.^{2} are effective.

Assembly of the apparatus. Tap water is passed through the electrode compartments when desalting, but distilled water is necessary for fractionation by electrodialysis.

When the apparatus is arranged for desalting, one centre compartment is used with a Permaplex anion-exchange membrane next to the anode compartment and a similar cation membrane next to the cathode compartment. To check that the membranes are correctly arranged the centre compartment is filled with distilled water and the current is switched on; there should be a small initial flow of current, dropping rapidly to zero. If, however, the membranes have been reversed, there will be a flow of current gradually increasing from a small initial value as the ions in the tap water are driven through the membranes into the centre compartment.

For electrodialysis extra compartments are added and the membranes are arranged in the sequence: cathode, anion membrane, cathode compartment, cation membrane, sample compartment, anion membrane, anode compartment, cation membrane, anode. In this way cations migrating into the cathode compartment are trapped by the anion membrane, and conversely for the cathode compartment. Alternatively, a series of cation membranes can be arranged on the cathode side and a series of anion membranes on the anode side. At the end of the run the farther compartments will contain a higher proportion of the faster-moving components of the mixture.

RESULTS

Preliminary experiments

The apparatus was assembled for electrodialysis as described, and 0·1 g. of sodium chloride was placed in the centre compartment. A current of 0·90 A was passed, and after 5 min. this had fallen to 0·02 A. The solution was removed and on evaporation no appreciable residue was obtained. The theoretical amount of electricity required to transport 0·1 g. of sodium chloride to the electrodes (assuming that all the current is carried by the ions of the salt) is 166 coulombs. If the average current throughout the run is taken to be 0·90 A, then 270 coulombs were consumed; this represents a maximum value, since the current fell steadily during the run. However, it is clear that the efficiency of desalting was high. Under the same conditions 0·5 g. of sodium chloride could be removed from the centre compartment in 30 min.

When 0·1 g. of glycine was placed in the centre compartment no current could be passed. When the glycine was replaced by 0·1 g. of glutamic acid, the whole of the glutamic acid was transported to the anode compartment in 10 min. at a current of 0·20 A. Conversely, 0·1 g. of arginine was transported completely to the cathode compartment in 5 min. at a current of 0·20 A.

Desalting of meat extract

After preliminary experiments of the type outlined above, a desalting curve for meat extract was constructed.

The meat extract (50 ml.) was placed in the apparatus arranged for desalting. Current was taken from the same tapping of the transformer (50 v) throughout the run. Portions (3 ml.) of the solution were removed for analysis, without affecting appreciably the resistance of the cell at that instant. The dry weight, nitrogen and ash content were determined on each sample and plotted, together with the resistance of the cell, against time. The current fell from an initial value of 0·56 to 0·10 A at the end of the run; the average current was about 0·40 A.

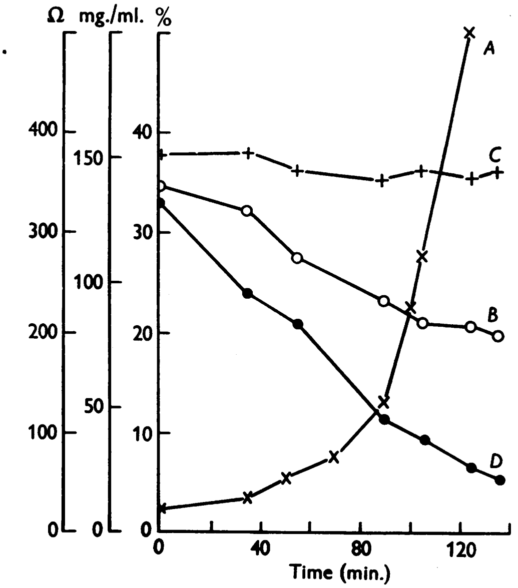

Fig. 2. Desalting of meat extract, *A*, Resistance (Ω); *B*, dry weight (mg./ml.); *C*, nitrogen (mg./ml. × 10); *D*, ash (%).

The curves obtained are shown in Fig. 2. The ash content has fallen from 33 to 5·7 %, and the nitrogen content has remained stationary. The sudden steep rise in resistance marks the approaching end of the run when a gradually increasing fraction of the current is being carried by organic ions. If the run is continued beyond this point, the recovery may fall from 60 % of the original material to less than 30 %.

Of the 6·93 g. of material taken initially, 3·94 g. (57 %) was recovered. Calculated from the initial and final percentages of ash, about 1·87 g. of inorganic material was removed, leaving 16 % of the organic material unaccounted for. This figure represents the maximum loss of organic matter, since the exact form in which the inorganic salts were present, before being converted into ash, is not known.

Table 1. *Fractionation of meat extract*

Cathode compartment	Centre compartment	Anode compartment
Sodium hydroxide, potassium hydroxide, ammonia, creatine, methylguanidine, trace of carnosine	Carnosine, creatinine, amino acids, creatine, guanidine, carnitine, hypoxanthine, inosine, an unidentified iminazole, a phosphate compound	Hydrochloric acid, phosphoric acid, lactic acid, unidentified organic acids, an unidentified guanidine compound, traces of amino acids

Fractionation of meat extract by electrodialysis

The apparatus was assembled for electrodialysis as previously described; with meat extract in the centre compartment the current was passed for a time considerably longer than that corresponding to the steep rise in the resistance curve (Fig. 2). At the conclusion of the run the contents of each compartment were identified by paper chromatography (see Table 1).

DISCUSSION

The ease with which any ionic species passes through the membranes will depend on its ionic mobility in the membrane, and in simple systems this can be expressed by its transport number. The transport number of cations in the cation membrane is high and that of anions is low, but the converse is true for the anion membrane (cf. *Permutit Bulletin*, Ion Exchange Membranes). It is upon this fact that the selective permeability of the Permaplex membranes depends. The quantity of electricity carried at any moment by a particular ionic species is proportional to the product czu, where z is the ionic charge, u is the mobility, and c is the ionic concentration, which is a function of the molecular concentration and the dissociation constant of the molecule. The fraction of the total current being carried by any particular ionic species depends upon the magnitude of this product relative to the sum of the products for the other ions in solution.

The amount of any substance being transported will therefore depend on its ionic concentration and mobility, relative to the other ions, both in the free solution and in the membrane. In general, these factors operate to favour the separation of inorganic from organic materials. However, electrodiffusion of ions through the membrane can increase the simultaneous diffusion of non-electrolytes (Lightfoot & Friedman, 1954), a factor tending to reduce the efficiency of desalting.

Practical applications

It is clearly demonstrated by the recovery and shape of the desalting curves that desalting in this apparatus is efficient, and it should be applicable to a wide range of natural mixtures. Considerable amounts of material, 10 g. or more, can be handled, in the space of a 2 hr. run, so that the apparatus is suitable for preparative work.

The type of fractionation by electrodialysis demonstrated in Table 1 should be a useful preliminary to chromatography, especially where organic acids are to be identified.

SUMMARY

1. The construction of a simple Perspex electrodialysis cell involving the use of Permaplex ion-exchange membranes or collodion membranes is described.

2. The apparatus can be used for desalting or for fractionation of complex mixtures by electrodialysis.

3. As an example of the use of the apparatus, meat extract containing 33 % of ash was desalted to an ash content of 5·7 % without appreciable loss of nitrogen.

The same meat extract was separated into an anionic, a cationic and a middle fraction by electrodialysis and the constituents were identified by paper chromatography.

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REFERENCE

Lightfoot, E. N. & Friedman, I. J. (1954). *Industr. Engng Chem.* **46**, 1579.