Short Communications

The preparation of ECTEOLA-celluloses of various capacities

In our work on the chromatographic purification of the antihaemophilic factor (AHF)[1] we used ECTEOLA-cellulose prepared from Whatman Standard Ashless Cellulose Powder by the method of PETERSON AND SOBER[2]. Several batches prepared in this way had capacities ranging from 0.36 to 0.42 mequiv./g. Although columns of these anion-exchangers worked satisfactorily only very limited quantities of AHF were actually bound. Eight grams of ECTEOLA-cellulose could bind no more AHF than the amount contained in 100 ml blood plasma which is a few mg at most. We therefore tried to increase the capacity of the ECTEOLA-cellulose. Using more NaOH or longer incubation at 0° for better mercerization (*cf.* PORATH[3]) was unsuccessful. Only processing ECTEOLA-cellulose (OH^-) of 0.38 mequiv./g a second time in exactly the same way resulted in an increase of the exchange capacity to 0.78 mequiv./g.

The AHF-binding capacity was not increased, however, or, at least, the recoveries were rather low, with the result that from this point of view working with ECTEOLA-cellulose of high capacity was not promising. At the same time the AHF-preparations obtained with this type of ECTEOLA-cellulose showed a greater density at 280 mμ, and thus contained more impurities. Because the aim of our work was to obtain AHF-preparations of the greatest possible purity, we then tried to prepare ECTEOLA-cellulose of low capacity. Halving the NaOH concentration proved ineffective. We then varied the ratio of epichlorohydrin:triethanolamine. In this way a series of ECTEOLA-celluloses of different capacities were prepared, ranging from 0.05 to 0.60 mequiv./g. Table I gives a survey of the results obtained.

TABLE I

Starting materials			*Capacity mequiv./g*
Cellulose material 30 g	*Epichlorohydrin ml*	*Triethanolamine ml*	
Cellulose	10	85	0.05
	15	40	0.14
	20	35	0.19
	22.5	32.5	0.27
	25	30	0.33
	30*	17.5*	0.36–0.42
	30	25	0.46
	35	20	0.60
	40	15	0.52
ECTEOLA-cellulose 0.38 mequiv./g	30*	17.5*	0.78

* Original method of PETERSON AND SOBER.

BOSCH *et al.*[4] only varied the amount of triethanolamine and thus obtained capacities ranging from 0.01 to 0.22 mequiv./g.

We started from 30 g of Whatman Standard Ashless Cellulose Powder, to which a cold solution of 30 g NaOH in 75 ml water was added in portions. The mixture was left standing overnight in the cold room, and the next morning the mixture of epichlorohydrin and triethanolamine was added. The ingredients were thoroughly mixed, and left at room temperature until a spontaneous reaction took place. After this had subsided the resulting product was washed and dried as described by PETERSON AND SOBER. In the first experiment of Table I, 85 ml triethanolamine was used erroneously instead of 45 ml. In the final experiment of that series it can be seen that

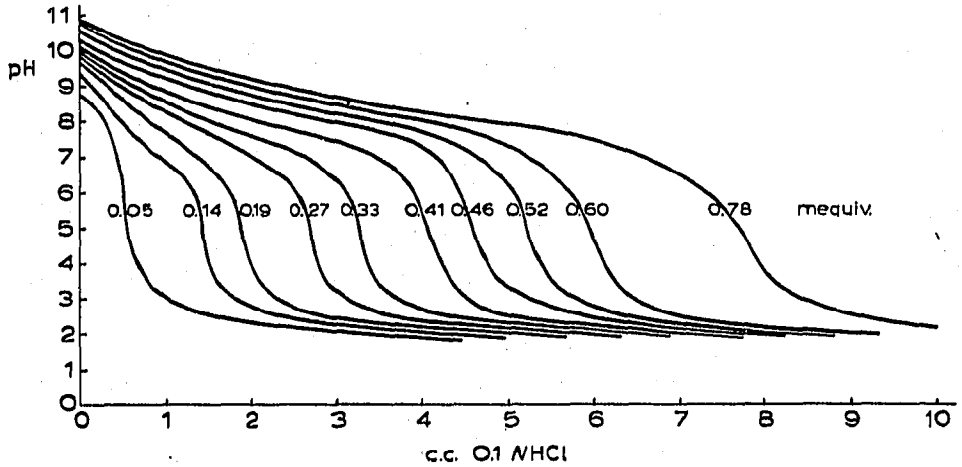

Fig. 1. Titration curves of several types of ECTEOLA-cellulose.

further increase of the ratio epichlorohydrin:triethanolamine only resulted in a lower capacity. This was to be expected.

The titration curves of the different types of ECTEOLA-cellulose are shown in Fig. 1. During these titrations we were struck by the very low exchange velocity in the pH-range 5.5 to 8.

The effect of different capacities on the chromatography of AHF will be described in another paper.

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