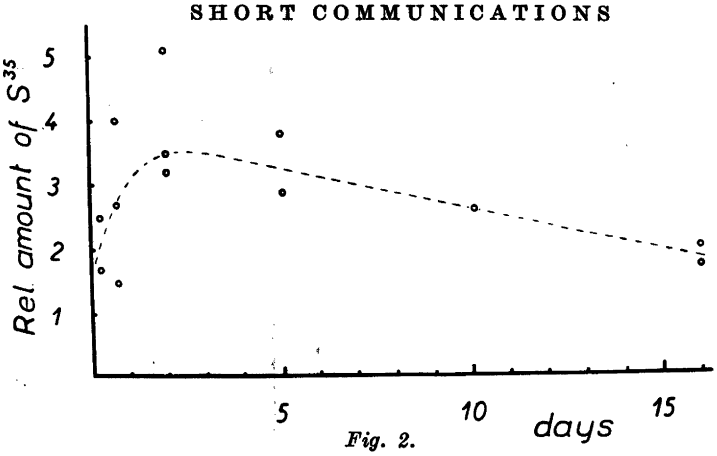

Fig. 2.

nic sulphur-containing compounds in the sections. It is therefore impossible, on the basis of the present investigation alone, to draw any conclusions with regard to the S^{35}-containing compounds in the aortic wall. The following facts have, however, been established by means of earlier investigations. (1) The greater part of the labelled sulphate disappears rapidly from the tissues [4,9]. (2) Sulphate-sulphur is not utilized in the synthesis of sulphur-containing amino acids [10,11]. (3) Good agreement is found if, after the administration of labelled sulphate, the incorporation of S^{35} in ester sulphates in the cartilage [4] and skin [12] of rats as determined on isolated chondroitin sulphuric acid is compared with the increase and decrease of S^{35} in these tissues [5,6], as estimated by quantitative autoradiography.

For these reasons it appears probable that, in the aorta as well, the greater part of the S^{35} present more than 24—48 hours after the injection of sulphate is in the form of ester-bound sulphur in chondroitin sulphuric acid. Thus, the curve in Fig. 1 reflects the exchange of the sulphate group in the chondroitin sulphuric acid of the aorta, the rate of which exchange seems to be moderate.

1. Meyer, K., and Rapport, M. M. *Science* **113** (1951) 596.
2. Dziewiatkowski, D. D. *J. Biol. Chem.* **189** (1951) 187.
3. Layton, L. L. *Cancer* **4** (1951) 198.
4. Boström, H. *J. Biol. Chem.* **196** (1952) 477.
5. Boström, H., Odeblad, E., and Friberg, U. *Arch. Biochem. and Biophys.* **38** (1952) 283.
6. Boström, H., Odeblad, E., and Friberg, U. *Acta pathol. et microbiol. Scand.* In press.
7. Jorpes, E., Odeblad, E., and Boström, H. *Acta Haematologica*, In press.
8. Odeblad, E. *Acta Radiol. Suppl.* **93** (1952).
9. Dziewiatkowski, D. D. *J. Biol. Chem.* **161** (1945) 723.
10. Tarver, H., and Smith, C. L. A. *J. Biol. Chem.* **130** (1939) 67.
11. Boström, H., and Åqvist, S. E. G. *Acta Chem. Scand.* **6** (1953) 1557.
12. Boström, H., and Gardell, S. *Acta Chem. Scand.* **7** (1953) 216.
13. Odeblad, E., and Boström, H. *Acta Radiol.* In press.

Received January 12, 1953.

A Small Cell for Electrolytic Alkoxylation of Furans

FRANZ LIMBORG
and NIELS CLAUSON-KAAS

Centrallaboratoriet, Sadolin & Holmblad A/S, Copenhagen, Denmark

To date 14 different 2,5-dialkoxy-2,5-dihydrofurans have been prepared by electrolytic alkoxylation of furans [1-7]. These alkoxylations were carried out in a cell with an outer and an inner cathode, containing about 300 ml of electrolyte [1].

The amounts of the various, analytically pure dihydrofurans, obtained in one run, varied from 11 to 96 g, the yields varied from 61 to 96 per cent. The dihydrofurans were all isolated by distillation.

We have now designed a small cell for alkoxylation of from 15 g down to less than 1 g of a furan. The cell, which only has one cathode, is filled with from 10 to 50 ml of electrolyte. The electrode distance is the same as in the larger cell (0.70 cm); but, since all the liquid in the small cell lies between the electrodes, the time for a single electrolytic preparation is reduced to about one third. The liquid is also cooled more efficiently than the liquid in the larger cell so that a larger current density can often be employed (about 4 instead of 2 ampere per dm^2). Hereby the time of electrolysis is further reduced.

The cell is especially useful for the preparation of small amounts of such dihydrofurans, as can be isolated by crystallization or be transformed without isolation into crystalline compounds.

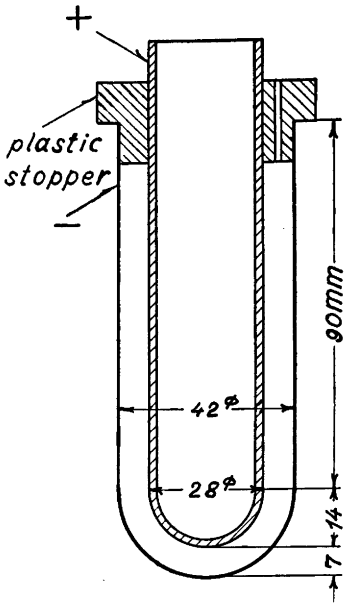

Fig. 1. Small cell for electrolytic alkoxylations.

Experimental. Fig. 1. gives a detailed design of the cell. The cathode consists of 0.5 mm sheet nickel, the anode is a glass tube covered with sheet platinum (welded on a metal form). The volume of the hemispherical part of the cell is 14 ml; the surface of the hemispherical part of the anode is 12 cm^2. During electrolysis the cell is placed in a bath of — 22° and connected with an ammeter and a coulometer of the ordinary domestic type.

In order to demonstrate the use of the cell for the preparation of small amounts of dihydrofurans a solution of 680 mg of furan (0.01 mole) and 150 mg of ammonium bromide in 10 ml of methanol was electrolyzed. 0.54 ampere hours (0.01 faraday) was passed through the cell (current 0.2—0.1 ampere, potential across the cell 4.2 volt, time of electrolysis 4.1 hours). After electrolysis 60 ml of *N*/10 hydrochloric acid was added and the mixture left standing overnight at room temperature. Hereby the dimethoxydihydrofuran formed was hydrolyzed to malealdehyde. 3.0 ml of phenylhydrazine and 5.0 ml of acetic acid in 40 ml of water were added. A yellow precipitate of malealdehyde bis-phenylhydrazone was immediately formed. After standing for 1 hour at room temperature the precipitate was filtered off, washed thoroughly with water and dried. Yield 1.84 g of crude hydrazone = 70 %; yield after crystallization from acetone-benzene 1.49 g = 56 %; m.p. 167—169° (Kofler stage, corr.); previously found [8] 171°. (Found: C 72.8; H 6.0; N 21.5. Calc. for $C_{16}H_{16}N_4$ (264.3): C 72.7; H 6.1; N 21.2.)

1. Clauson-Kaas, N. (to Kemisk Værk Køge A/S). Belg. patent 500356 (1951).
Clauson-Kaas, N., Limborg, F., and Glens, K. *Acta Chem. Scand.* **6** (1952) 531.
2. Clauson-Kaas, N., Limborg, F., and Dietrich, P. *Acta Chem. Scand.* **6** (1952) 545.
3. Clauson-Kaas, N., and Limborg, F. *Acta Chem. Scand.* **6** (1952) 551.
4. Clauson-Kaas, N. *Acta Chem. Scand.* **6** (1952) 556.
5. Clauson-Kaas, N. *Acta Chem. Scand.* **6** (1952) 569.
6. Elming, N. *Acta Chem. Scand.* **6** (1952) 572.
7. Clauson-Kaas, N., and Tyle, Z. *Acta Chem. Scand.* **6** (1952) 667.
8. Clauson-Kaas, N., and Fakstorp, J. *Acta Chem. Scand.* **1** (1947) 210.

Received January 12, 1953.