Table 1. The contents of sodium and potassium in the endo- and perilymph.

	Na mmoles	K mmoles
Endolymph	2.80	0.60
Perilymph	2.47	0.37

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Syntheses in Capillary Tubes

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For several years the students at the Institute of Chemistry, Helsinki University, have carried out several syntheses on a micro scale, using as textbook A. A. Morton's "Laboratory Technique in Organic Chemistry". Experiment on a micro scale included in this book have chiefly been devised by A. Fuchs¹. The reactions and the purification of substances are carried out in a capillary tube about 2 mm in diameter. Fuchs freed the crystals from the mother liquor by centrifuging the contents of the reaction tube to the bottom and sucking off the liquid with a capillary pipette. However, it is not possible to remove all the liquid in this way. This is a serious drawback, since it is known that complete removal of the mother liquor is a necessary condition for the purification of substances by crystallization.

The present author has devised some improvements to Fuchs' technique, that make the micromanipulation easier and more effective. The most noteworthy of them is the manner of removing the mother liquor from the crystals.

Fig. 1. Explanation in the text.

This is a general course of a synthesis with modifications:

A capillary tube, 2 mm in diameter is drawn out from 10 mm glass tubing. 6 cm long pieces of this capillary tube are sealed at one end. The small "test tubes" so made are here called reaction tubes.

Liquid substances are best introduced by means of 3 cm long pieces of 1 mm capillary tube (the same kind of capillary tubing as is used in melting-point determinations). Each piece of tube is used for filling one substance only, a new piece being taken for each substance. When a liquid is touched with one end of a capillary tube, it rises spontaneously in the tube. The height of the liquid column in the capillary tube is a measure of the quantity of the liquid. Any excess of liquid is removed by touching a piece of filter paper with the lower end of the capillary tube. To transfer the substance from the filling capillary to the reaction tube, the former is placed half-way into the latter (Fig. 1 a). The liquid can be shaken to the bottom of the reaction tube by jerking the tubes by hand. When all the necessary substances are in the reaction tube, it is sealed and heated in a metal block for the time required for the reaction. When the reaction is complete, the tube is opened. At this stage the reaction mixture can be stirred with a capillary stirring rod to promote the

crystallization. The reaction tube is then drawn to a fine capillary near its open end (Fig. 1 b). The capillary is cut off at the place marked in Fig. 1 b. A 3 cm long piece of 3 mm tubing is sealed at one end and is heated at one point in a micro flame to produce a small recess. The reaction tube is then placed in this filtering tube, the capillary end inwards; the filtering is achieved by centrifuging the liquid away from the reaction tube (Fig. 1 c). On centrifuging, the crystals usually remain at the closed end of the reaction tube; if they come loose, they are held back by the capillary end, which functions as a filter. If the precipitate is very fine, it is better to use an asbestos filter (Morton, p. 166). If some crystals reach the capillary end of the reaction tube, they can be brought to the bottom by dropping the reaction tube through a long glass tube against some hard object.

For recrystallization of a substance, a little solvent is taken up in the filling capillary. The reaction tube is gently heated, and its capillary end is put into the filling capillary (Fig. 1 d). On cooling, the reaction tube sucks the solvent in. Washing liquids and precipitating agents can be introduced in the same way. After the solvent has been centrifuged to the bottom, the crystals are dissolved by heating the tube in a metal block (not over a naked flame!). It is best to let part of the substance remain undissolved; it then initiates crystallization on subsequent cooling. If a supersaturated solution does not begin to crystallize, a minute amount of liquid is allowed to drain out of the capillary end. On the outer surface of the capillary the solution can be brought to crystallize by rubbing with a capillary glass rod. The crystallization then propagates through the capillary part into the reaction tube.

The crystals are then freed from the liquid by centrifuging as above. The drying of the crystals is best accomplished by heating the reaction tube in an evacuated test tube (Fig. 1 c). The vacuum is released a couple of times during drying. After drying the melting-point of the substance is determined in the same tube. The crystallization can then be repeated if necessary.

For sublimations, the capillary tube is heated in the same way as in melting-point determinations (Kajola's apparatus²). The sublimation is greatly accelerated if a minute hole is left at the bottom

of the capillary tube. Since the air in the melting-point apparatus is in rapid motion, a slow current of air is produced through the capillary tube; the air current conveys the vapours of the substance to the colder part of the tube, where they are condensed (Fig. 1 f).

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isoThiocyanates IX. The Occurrence of Ethyl isoThiocyanate in Nature

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The naturally occurring isothiocyanate-containing glycosides can, for practical reasons, be classified in two groups according to whether their isothiocyanates are volatile with steam or not. Within the former group the occurrence of allyl, (+)-*sec*-butyl, benzyl and β -phenylethyl isothiocyanate was convincingly established before these studies were initiated two years ago. Since then, we have proved 3-butenyl isothiocyanate to be derivable from a glycoside of, a.o. *Brassica napus* L.¹, and demonstrated by isolation the wide-spread occurrence of isopropyl isothiocyanate in the family *Cruciferae*². To these findings we now wish to adduce experimental evidence for the presence in nature of a glycoside containing ethyl isothiocyanate, the occurrence of which has not been suggested previously.

In the course of a paperchromatographic scanning of a large number of seed-samples for their contents of volatile isothiocyanates, the results of which have been partly published³, a rather unique spot with an R_F -value⁴ of 0.15 was noticed from the seeds of *Lepidium Menziesii* DC. This value is in accord with that of authentic N-ethylthiourea, suggesting the presence of ethyl isothiocyanate in the glycoside of the seed in question. Unfortunately, the small amount of seed available at that time did not allow substantiation of this assumption by isolation. Through the courtesy of