

## Apparatus for Simple Laboratory Batch Distillation

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A rugged and versatile setup for distillation of charges up to 5 liter volume is described. The setup is intended mainly for work in organic chemical and biochemical laboratories. It has been in daily use for seven years and has been tested in four different laboratories. All simple laboratory distillations, at pressures above 0.1 mm, can be carried out in this, or one of the two other setups, described previously by us (*Acta Chem. Scand.* 8 (1954) 1579).

This communication describes a distillation setup intended for (a) the purification of solvents (b) the removal of solvents and (c) the distillation of amounts larger than 100 ml of liquid organic compounds.

The setup (Fig. 1), which is mounted at the end of a laboratory bench, is especially simple and rapid to operate. After putting the distillation flask in place, outside of the water bath, the steel support rod is lifted, and the whole apparatus turned, so that the distillation flask may be immersed in the bath. *Since the three clamps supporting the glass part of the apparatus are directly aligned, there is no risk of breakage, or of leakage at the ground joints, being caused by the strain of mounting.*

Owing to the large diameter of the tube leading from the distillation flask to the condenser, distillations can be carried out rapidly and at low pressure. Table 1 shows three representative distillations illustrating the capacity of the setup.

The construction and use of the setup is described in detail below.

*The distillation and the receiver flasks have either glass hooks (sizes 100, 250, 500 and 1 000 ml) or metal holders (sizes 2, 3 and 5 liters) permitting attachment of springs. When distilling under vacuum no springs are used to hold the distillation flask, but the flask is held by hand until immersed in the water bath. When solvents are distilled*

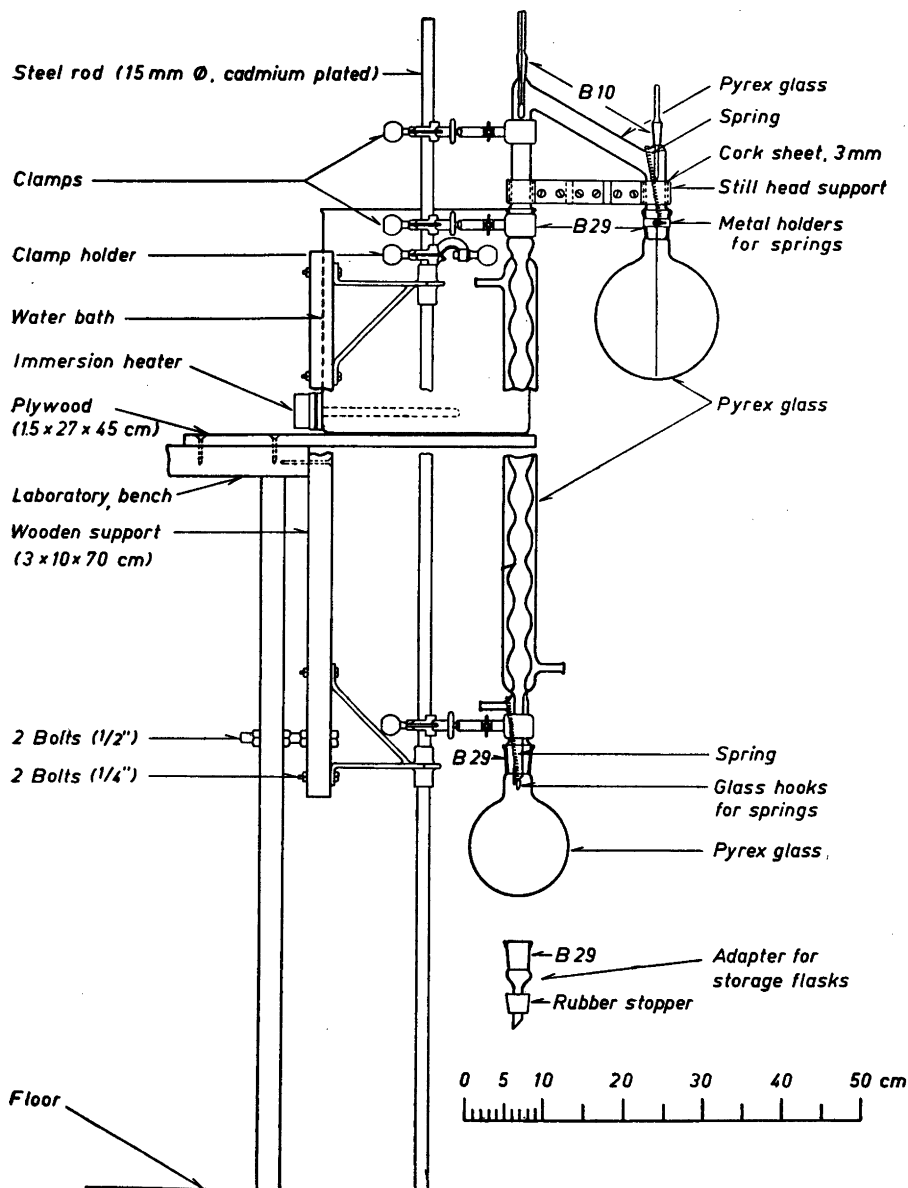


Fig. 1. Setup for simple distillation of up to 5 liters, viewed from rear.

under atmospheric pressure directly into storage flasks, an adapter as shown in Fig. 1 should be used.

The still support (3 mm iron or brass) is made of three parts to prevent breakage of the head due to strain. It is fixed to the glass by first tightening the two pairs of screws

Table 1. Representative distillations. In the case of the distillation of water, the still head was insulated with a cloth and the receiver flask cooled with ice.

Compound	B. p. at 760 mm °C	Charge ml	Yield of distillate ml	Distilla- tion temp. °C	Bath temp. °C	Back pressure mm Hg	Distilla- tion time min.
Acetone, crude	56	2 000	1 974	55-61	81-94	757	72
Water	100	1 000	940	36-38	75-78	26-29	64
1-Methyl- naphthalene	245	100.0	98.2	58-59	87	0.1	22

at the ends, and then the pair in the middle. The still head can carry a distillation flask weighing 5 kg without its breaking.

Suitable clamps for supporting the glass part of the setup are utility clamps (large size, rubber sleeve) from *Fisher Scientific Company*, Pittsburgh, U.S.A.

The water bath is designed for distillation flasks up to 5 liters. It is heated by a 1 500 watt immersion heater connected with the network through a bimetal regulator. In order to minimize evaporation of water, the bath is covered with a lid, slotted to receive the neck of the distillation flask. The height of the distillation flask is adjusted by use of the clamp holder, which is attached directly above the upper support for the steel rod. In general, the distillation flask should be immersed up to its neck in the water bath in order to prevent foaming. If required, the flask can easily be shaken by hand while in the bath during a distillation.

For the distillation of very high-boiling liquids, the water bath is replaced by some other heating device.

The supports for the steel rod are 40 mm wide and screwed symmetrically on to the wooden support. The wooden support is fixed to the laboratory bench in such a way that the center line of the support is 42 cm from the front of the bench. The plywood plate carrying the water bath is fixed in line with the front of the bench.

Rubber tube connections to cooling water and vacuum pumps should be mounted so that they do not disturb the lifting and turning of the apparatus.

Cleaning of the still head and the condenser is best accomplished by sucking a suitable solvent once or twice from a 50 ml beaker through the socket of the joint on to which the distillation flask fits.

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