Distillation

Distillation is a process by which a mixture of substances are separated thermally. It utilises the evaporation of the liquid sample by heating, and then condensation of its vapours by cooling and subsequently collected in a receiver.

Theoretical background

Distillation (Latin; destillare, to drip down) is the evaporation of a liquid consisting of any number of components and the subsequent condensation of the vapour, to give the distillate. Fractionated distillation is the thermal separation of at least two distillates on the basis of them having different boiling points.

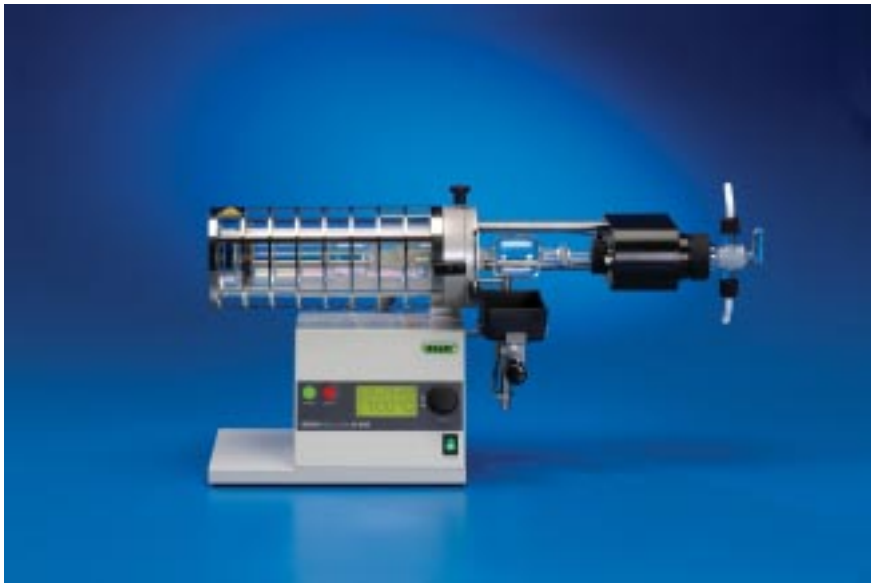
Method

Materials which are temperature sensitive must be treated with care to avoid decomposition. Therefore, is it important to have gentle, indirect heating. The working principle of the BÜCHI Glass Oven B-585 utilises a glass tube which is coated with a transparent and electrically conductive layer. This way of heating delivers moderate heat radiation, which is evenly distributed across the entire heating surface and facilitates heat entry of the material without direct contact of the heat source, thus minimising thermal decomposition. The glass on which the layer is coated on has good heat conductivity and high thermal capacity, therefore the majority of the heat generated is radiated inward.

The electrically conductive layer provides fast heating to the oven and since 75% of the heat is supplied to the material is by radiation and only 25% by convection, sample heating is quick. The oven heats up to 300°C and the internal temperature is monitored by an integrated sensor. And since the glass oven is completely transparent, visual observation of the sample is possible, thereby any decomposition can be observed and actions can be taken.

Two type of distillation are possible with the glass oven: simple distillation and fractionated distillation. Simple distillation is accomplished by having one ball in the oven and another ball outside, being cooled. Fractionated distillation is starts with up to balls in the oven, and one outside. As each fraction is collected, one of the balls in the oven is shifted outside to collect the next fraction. The process of simple distillation can be compared to that of a rotary evaporator, but on a much smaller scale.

BÜCHI Glass Oven B-585



Simple distillation with the B-585 ball tube

The distillation is achieved by placing the sample in a glass ball, positioned inside the oven. A second ball, which is connected to the first one, is placed outside the oven and is cooled using a cooling device. The oven is then heated to enable distillation. The evaporated solvent or product is condensed by the cooling device, preventing it from being sucked into the pump and subsequently, the atmosphere. Rotation of the ball tube optimises heat transfer and reduces boiling of sample.

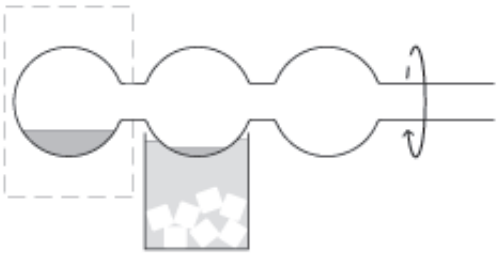
The heating temperature, which is set at around 10°C to 40°C above the boiling temperature of the sample (depending upon type of material and pressure), is regulated by a vacuum controller, are the relevant. The heating temperature is typically set at around 10°C to 40°C above the boiling temperature of the sample (depending on type of material and pressure). A vacuum controller may be used to regulate process parameters for distillation.

For thermally unstable substances, it is preferable to use low vacuum and for solvents with very low boiling point, it is advisable to use a vacuum system with a secondary condenser in order to condense residual solvents.

Fractionated distillation

Mixtures of products can be separated as long as there is a difference of 20°C or more between the individual components. As many balls as components in the mixture can be interconnected (but only a maximum of four balls may be placed in the oven.

Example: A three-component mixture needs 1 end-ball and 2 mid-balls.



The mixture that is to be separated is placed into the end ball. All the balls except for the last empty glass ball are inserted into the oven chamber, with the last glass ball cooled in the cooling device (with ice or dry ice-alcohol mixture).

The temperature of the oven then is increased until distillation of the component with lowest boiling point begins.

Once the distillation of this fraction is complete, i.e. volume does not increase in the received fraction (in the cooled ball), the distillation is halted.

The next ball is then pulled from the oven and the temperature is increased. Thus the next high-boiling component is distilled and deposited in the second cooled ball. This procedure repeats itself until all balls are filled with fraction.

BÜCHI SWITZERLAND

Quality in your hands

BÜCHI Labortechnik AG
Postfach
9230 Flawil
Schweiz
Tel. +41 71 394 63 63
Fax +41 71 394 65 65
buchi@buchi.com
www.buchi.com

BÜCHI Labortechnik GmbH
Postfach 10 03 51
45003 Essen
Deutschland
Freecall 0800 414 0414
Fax +49 201 237 082
deutschland@buchi.com
www.buechigmbh.de

BÜCHI Labortechnik GmbH
Branch Office Netherlands
Postbus 142
3340 AC Hendrik Ido Ambacht
The Netherlands
Tel. +31 78 684 94 29
Fax +31 78 684 94 30
netherlands@buchi.com
www.buchi.com

BÜCHI Italia s.r.l
Centro Direzionale, Milano Fiori
Pal. A-4, Strada 4
20090 Assago (MI)
Italia
Tel. +39 02 824 50 11
Fax +39 02 57 51 28 55
italia@buchi.com
www.buchi.it

BACC **BUCHI**
ASEAN Competence Center
c/o Becthai Bangkok
300 Phaholyothin Road
Samsennai, Phayathai
Bangkok 10400
Thailand
Tel. +66 1 829 49 10
bacc@buchi.com
www.buchi.com

BUCHI Analytical Inc.
19 Lukens Drive
New Castle
Delaware 19720
USA
Tel. +1 302 652 3000
Fax +1 302 652 8777
us-sales@buchi.com
www.buchi-analytical.com

BUCHI Analytical Ltd.
Link House
Stakehill Industrial Park
Middleton
Manchester M24 2RH
Great Britain
Tel. +44 161 654 60 60
Fax +44 161 643 75 10
uk-sales@buchi.com
www.buchi-analytical.co.uk

Nihon **BUCHI** K.K.
7F Kojima Bldg. 2-11-10 Ueno
Taito-ku
Tokyo 110-0005
Japan
Tel. +81 3 5807 5599
Fax +81 3 5807 5598
nihon@buchi.com
www.buchi.com

BUCHI Sarl
5, rue du Pont des Halles
Z.A. du Delta
94656 Rungis Cedex
France
Tél. +33 1 56 70 62 50
Fax +33 1 46 86 00 31
france@buchi.com
www.buchi.fr

BUCHI SMP Services Private Ltd.
201, Magnum Opus
Shantinagar Industrial Area
Vakola, Santacruz (East)
Mumbai 400 055
India
Tel. +91 22 569 89 450
Fax +91 22 569 89 452
smplisp@vsnl.com
www.buchi.com

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