Centrifugal Evaporation of Organic Solvents from a Series of Solutions

J. A. ONTKO and R. F. JONES, Department of Biochemistry and Instrument Division, University of Tennessee Medical Units, Memphis

Abstract

A centrifugal vacuum evaporator designed for the evaporation of organic solvents from a series of solutions has been fabricated and tested. The rotor is driven by a spinning magnetic field resulting from rotating magnets mounted on a wheel connected to the drive shaft of an electric motor beneath the vacuum chamber. The spinning magnetic field produces eddy currents in an aluminum disc in the vacuum chamber, thus rotating the disc and the attached rotor. A variety of organic solvents were readily evaporated in this device. Auxiliary heating was used to increase the evaporation rate of certain solvents. In this evaporator organic solvents are removed from a series of solutions simultaneously, at low temp, and in a partial vacuum, thus minimizing or preventing solute degradation by heat and oxygen.

Introduction

The evaporation of organic solvents from a series of solutions is an indispensable process in a wide variety of procedures in analytical chemistry. Problems associated with such a process include the explosive nature of many organic solvents, the bumping and foaming of liquids during evaporation, the lability of the compounds undergoing concentration, particularly in the case of biological preparations, and, in many cases, the multitude of samples which require evaporation.

Two general methods for the evaporation of such samples are in widespread use. One procedure involves the use of a heating bath (1), usually a metal block (2), and the other utilizes some type of rotary evaporation under reduced pressure (3,4). With the heating block, solvents may be removed from a number of samples simultaneously. When flammable solvents are undergoing evaporation, such a device may be somewhat hazardous although it can be placed in a hood and constructed to reduce the possibility of explosion. However, solutes are heated to the boiling point of the solvent in a heating block and are subjected to even higher temperatures when the evaporation is nearly complete. Labile compounds may therefore undergo partial or complete degradation. Also, occasional losses may result from violent bumping due to superheating (1,5), or foaming which is usually associated with low surface tension (5,6). The rotary evaporator, on the other hand, can be operated under reduced pressure at mild temps, in greater safety from explosion and with less foaming. However, the rotary evaporator accommodates only one sample at a time, and this is a severe limitation when a large number of samples need to be evaporated.

A device to alleviate the above problems has been fabricated. In this laboratory it is frequently necessary to evaporate organic solvents from a series of solutions at some stage of processing. These solutions are often in test tubes, or in containers from which aliquots are readily transferred to test tubes, when the solvent needs to be either partially removed to concentrate the solution or completely removed, usually to redissolve the sample in a different solvent. This evaporator was constructed to evaporate organic solvents simultaneously and directly from such a series of solutions in test tubes. It utilizes the principle of centrifugal vacuum evaporation, introduced by Greaves (7) for drying materials from the frozen state. This device resembles a commercially available lyophilizer (Bio-Dryer, Vir-Tis Co.) but is constructed with the basic guideline of avoiding sources of possible explosion when flammable organic solvents are undergoing evaporation. Throughout more than a year of extensive use, many different organic solvents have been safely and reliably removed from various solutions in this evaporator. This device, described herein, consists of a small centrifuge in a chamber which can be evacuated. The samples in test tubes are placed in the centrifuge rotor which is of simple construction. During evaporation, which proceeds readily when the chamber is evacuated, the high gravitational force prevents loss of solution from foaming or bumping. The temperature of the solution decreases during solvent vaporization. Evacuation greatly decreases the exposure of solutes to oxygen. Solute molecular alterations potentially induced by oxygen and heat are therefore minimized or eliminated.

Materials and Equipment

The power mechanism for driving the rotor is completely isolated from the centrifuge chamber (Fig. 1). This important feature greatly reduces the possibility of explosion. The driving mechanism consists of a 1/6 hp electric motor mounted vertically beneath the centrifuge chamber. The vertical motor shaft acts as the axle of an aluminum wheel. Three 6-oz horseshoe magnets, 30-lb pull, attached to this wheel (Fig. 2), therefore rotate in a horizontal plane. The base of the vacuum chamber (Fig. 1) is stainless steel which has low magnetic permeability and high electrical resistivity. This allows transmission of the rotating magnetic field into the vacuum chamber. The rotating magnetic field passes through a horizontal aluminum disc in the bottom of the vacuum chamber producing eddy currents and a rotating force in the disc. Aluminum has high electrical conductivity and low magnetic permeability. The high conductivity of the aluminum is desirable since it allows high eddy currents to flow, thereby producing the rotating torque, but the low permeability is undesirable since it does not give rise to a high flux density. A steel disc, attached to the top of the aluminum disc, serves as a magnetic flux return plate. This affords a higher flux density in the aluminum which results in a stalled torque approximately twice that of the aluminum disc alone. A vertical shaft attached to the rotating disc supports the centrifuge rotor which therefore spins at the same speed as the disc. The rotor, shaft and disc rest on a single, 0.25 in. ball thrust bearing. A variety of rotors have been fabricated to accommodate tubes with different dimensions. The largest rotor constructed for this evaporator holds fourteen 50 ml centrifuge tubes which, when spinning, have a capacity of 30 ml each. A 500w tubular heating element in the centrifuge chamber may be used to accelerate evaporation. This is useful for concentrating relatively large volumes of solvent, that is, when the tubes are filled to maximum capacity, and for evaporating solvents which have a high boiling point or a high heat of vaporization. The heating element, when used, is only turned on after evacuation of the vacuum chamber.

Although Plexiglas is attacked by certain organic solvents, extensive use of this evaporator has not resulted in noticeable damage to the wall or ceiling of the vacuum chamber, probably because the solvent vapors are at a low concentration in the evacuated system. Although it is not known if static electricity might accumulate sufficiently on the Plexiglas to present a hazard, the wall and top of the vacuum chamber were lined with aluminum screen as a precaution. This screen and the metal parts of the evaporator are at the same potential since all are grounded. To spot seal the screen to the Plexiglas, a solution of Plexiglas in ethylene dichloride was used. To allow better visual observation of the solvent meniscus with or without the aid of a stroboscope, a window was cut in this screen.

Although a glass vacuum chamber would also allow visual observation, Plexiglas was employed since it is not only less fragile and therefore safer, but also more easily procured, fabricated and sealed to the base plate.

The plastic vacuum chamber was attached to the stainless steel base plate with screws at 1.75 in. intervals. A silicone rubber adhesive cement was used to promote to tight seal between the Plexiglas and the base plate. On the other hand, the Plexiglas cylindrical vacuum chamber may be attached to the base plate with a split-rubber tubing gasket, coated with vacuum grease. In this case, the cylindrical chamber is readily removable. The Plexiglas adjacent to the heating element was protected from heat by a semicircular polished aluminum reflector, 5 in. high. To remove the possibility of heat-induced damage of the Plexiglas wall of the vacuum chamber, the voltage supply to the heating element, when used, was controlled to prevent the temperature of the Plexiglas from exceeding 50C.

The panel of the lower motor chamber of the evaporator contains the control knob of a variable transformer for regulating the voltage supply to the heating element, switches for a vacuum pump and the electrical motor which rotates the magnetic field, and a vacuum release valve to allow entry of air to the system when desired, usually after the evaporation is complete.

With the aid of a stroboscope, it was observed that the motor was turning the magnets at a speed of 1770 rpm and the rotor was spinning at 1600 rpm at atmospheric pressure and at 1695 rpm at either water aspirator or vacuum pump pressure. The actual slippage or loss in transmission of this type of magnetic drive was therefore only about 10% in air and 4% when the rotor was in a partial vacuum, 2-17 mm Hg. The difference between the rotating speed of the disc in the vacuum chamber and the speed of the magnets below this chamber—a difference which is largest during acceleration—does not build up an electric charge since there is no charge accumulation across a conductor. All rotating parts are at ground potential since they are constructed of metal and connected to the metal base plate which is connected to an earth ground.

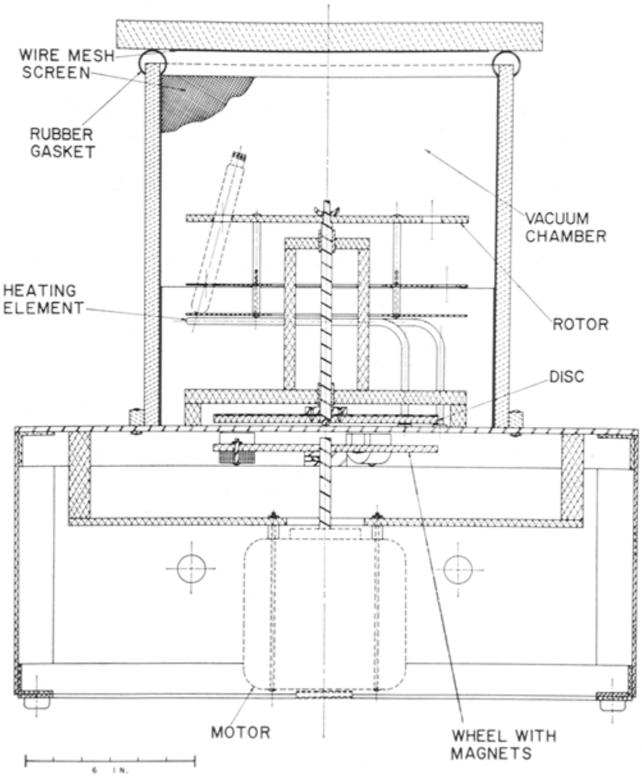

Fig. 1. A vertical cross section of the centrifugal vacuum evaporator. The base plate is 304 stainless steel, 0.15 in. thick. The rotor was painted black to absorb radiant heat. A thermometer was mounted in the vacuum chamber to monitor the temp although the heat distribution in the vacuum chamber is not uniform when the heating element is used. The clear, colorless Plexiglas wall of the vacuum chamber is 0.5 in. thick, and the ceiling of this chamber is much thicker (1 in.). The increased weight of the ceiling aids in the formation of an airtight seal at the beginning of the evacuation. A wire connected to the top of a screw through the Plexiglas ceiling was used to ground the aluminum wire screen attached to the ceiling. Small holes were bored in the disc where necessary for balancing. This disc is 0.25 in. thick, 2024-T4 aluminum and its diameter is 8 in. The top side of the disc is covered with a steel flux return plate, 0.0625 in. thick. The rotation of the vertical shaft connected to this disc is guided by two porous bronze bushings. Not visible in this cross section is a metal vacuum line, 0.25 in. diameter, leading from the base of the vacuum chamber through, and to the outside of, the lower housing containing the motor, magnets and circuitry.

Results and Discussion

To determine the rates of evaporation of various organic solvents in this evaporator, screw-cap test tubes, 20-ml capacity, were placed in a 20-tube rotor designed for this test tube size. These tubes, at a 13° angle in this rotor, have a capacity of 14 ml when centrifuged. Ten tubes, each containing 10 ml of a particular solvent, were placed in the rotor, centrifuged, and the time for complete evaporation under reduced pressure was determined (Table I). A trap of dry ice and methyl cellosolve was used to condense vapors and a manometer was placed in the vacuum line to monitor the pressure. A variety of organic solvents were readily evaporated under these conditions. The use of a water aspirator for evacuating the system resulted in a pressure of 17-18 mm Hg when the solvent was nearly gone. Although chloroform and methanol have similar boiling points, chloroform evaporated much faster than methanol. This reflects the relatively high heat of vaporization of methanol. The time of the evaporation was reduced only 5-10% by replacing the water aspirator with a vacuum pump (Table I). The pressure, when

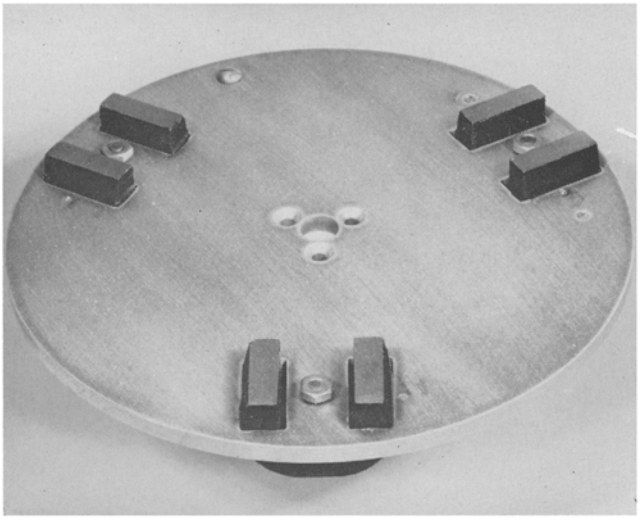

FIG. 2. The orientation of the magnets mounted on the aluminum wheel. This wheel has a thickness of 0.25 in. and a diameter of 8 in. It is 2024-T4 aluminum. The screw in the back left and the holes on both sides of the right magnet were added to balance the wheel after mounting the magnets. This wheel should be mounted as close as possible to the base plate (Fig. 1).

the vacuum pump was used for evacuation, was about 2 mm Hg when the solvent was nearly gone. The evaporation of chloroform and methanol proceeded rather slowly. However, the use of auxiliary heating increased the rate of evaporation markedly (Table I). Thus, even though the heat transfer from the heating element to the solvent is inefficient in this evaporator under reduced pressure, such a heat source is an important factor in reducing the evaporation time.

The advantage of using a vacuum pump in place of a water aspirator to decrease the evaporation time was minimized by the more rapid cooling of the solvent when a vacuum pump was used (Table II). If heat is not supplied, a liquid undergoing evaporation cools off rapidly as molecules with higher energy enter the vapor phase. The vapor pressure of a liquid diminishes as its temp decreases and therefore if the external pressure is constant, the evaporation decelerates. A concomitant decrease in the pressure in the system would tend to reverse this decrease in evaporation rate. The gradual decrease in the pressure in the system, however, was not sufficient to completely reverse the deceleration of the evaporation except in the early stage of evacuation when the water aspirator was used (Table II).

When diethyl ether or petroleum ether were evaporated, using the water aspirator, the trap condensed 75–85% of the solvent vapors. When the water aspirator was used without the aid of a trap in the system, the evaporation time of diethyl ether and petroleum ether was 19 and 21 min, respectively, as compared to 17 and 19 min, respectively, when a trap was present (Table I). Under these conditions, the use of a trap reduced the evaporation time by only 10%, indicating that solvent vapors were removed rather effectively by the water aspirator.

TABLE I

The Evaporation Time of Various Organic Solvents in the Centrifugal Evaporator

Solvent	Boiling Point or Range, °C	Evaporation Time, Min [a]		
		Water Aspirator	Vacuum Pump	Vacuum Pump with Heating [b]
Diethyl ether	34.6	17	16	
Chloroform	61.3	48	45	31
Petroleum ether	37–53	19	17	
Skelly B	62–70	28	25	18
Methanol	64.7	124	116	69

[a] The time required for the complete evaporation of 10 ml of solvent in each of 10 tubes. A trap of dry ice in methyl cellosolve was used.
[b] With 87 v supplied to the heater.

TABLE II

Effect of Evacuation Pump Type on the Rates of Cooling and Evaporation of Diethyl Ether in the Centrifugal Evaporator

Two tubes, each containing 10 ml of diethyl ether, were centrifuged and the system was evacuated for the indicated periods. The motor which spins the magnetic field was stopped 30 sec before the end of the evaporation period, indicated below, to allow the rotor to slow down. At the desired time, the line pressure was recorded, the vacuum release valve was opened and the pump was turned off. The rotor was nearly stopped at this time. The temp and vol of the solvent were then measured.

Time, Min	Water Aspirator			Vacuum Pump		
	Temp [a] °C	Vol ml	Pressure mm Hg	Temp [a] °C	Vol ml	Pressure mm Hg
0	26	10.0		26	10.0	
2	22	8.8		0	6.1	80
4	11	6.6		−21	3.2	7
6	3	4.4	102		2.0	4
10		1.3	26		0.6	3

[a] When the sample volume was appreciably less than 3 ml, the temp as measured was not representative due to warming of the solvent by the introduction of air and the thermometer bulb.

The effect of reducing the number of tubes containing solvent on the evaporation time in the centrifugal evaporator was also tested. With all solvents tested, namely diethyl ether, petroleum ether, Skelly B and chloroform, it was observed that the evaporation of solvent in two tubes, each containing 10 ml of solvent, required 20% less time than the evaporation of this volume of solvent in each of ten tubes. This comparison was made with a trap, containing dry ice in methyl cellosolve, in the system and without auxiliary heating.

Tubes with an equal volume of the same solvents should be centrifuged opposite each other for balancing. When each tube contains the same initial volume of the same solvent, the course of evaporation may be followed visually. When the rotor is spinning, the meniscus is visible through the cylindrical Plexiglas housing if eight or more tubes are evenly spaced in the rotor. However, when fewer tubes are spinning, the meniscus is not clear during centrifugation. Also, near the end of the evaporation, when small amounts of solvent remain, the meniscus is not readily observed regardless of the number of tubes undergoing centrifugation. Under these conditions, the use of a stroboscope provides a clear image of the solvent level in the spinning tubes. On the other hand after the initial period of the rapid evaporation, if the motor is turned off long enough to allow the rotor to slow down almost to a stop, the solvent meniscus becomes clearly visible, and such momentary reduction in centrifugation does not cause solvent loss from bumping.

The residues, after solvent centrifugal evaporation, are concentrated in a small spot on the side of the tube, near the bottom. This is of some aid in subseqently redissolving the solutes in a different solvent, which is often necessary in processing the materials of interest.

ACKNOWLEDGMENT

Mr. William A. Frase assisted in the construction of the centrifugal vacuum evaporator described herein.

This investigation was supported by U. S. Public Health Research Grant GM-07467.

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[Received September 25, 1964—Accepted December 10, 1964]