





<u>R</u>adially <u>A</u>ccelerated <u>V</u>ariable <u>E</u>ngine



Introduction

The CycloGraph system is a centrifugally accelerated device for performing preparative chromatographic separations. The device spins a layer of adsorbent material coated as a flat ring on a glass backing. A solvent pump is used to apply the sample and mobile phase to the center of the



spinning adsorbent ring. The centrifugal action accelerates the flow of the mobile phase through the adsorbent, separating the sample components as circular bands.

The mobile phase elutes continuously into a specially shaped collection channel inside the body of the instrument. Component bands are collected manually in test tubes or optionally by an automated fraction collector (not included.) Separations occur quickly, usually within 20 minutes, versus the typical 90 to 120 minutes for preparative Thin Layer Chromatography (TLC) or Column Chromatography.

Principle of Operation

The stationary phase for the CycloGraph instrument is called a Rotor. A Rotor is similar to a preparative TLC plate but it is round instead of rectangular. The round glass support of a Rotor has a hole in the center so that it can be mounted on the motor of the CycloGraph. The motor will spin the Rotor at 720 RPM (see specifications.) The adsorbent material of the Rotor is coated as a ring that does not extend into the center hole of the glass. This clear zone of glass at the center is where the sample and mobile phase will be applied.

The sample is applied first as a solution by way of the solvent pump or a hand held syringe. The spinning action of the Rotor will force the sample liquid to flow as a uniform round band on the inner edge of the adsorbent ring. The solvent pump is then switched to the mobile phase chosen to separate the components of the sample. The spinning action of the Rotor, and to a lesser degree the pump flow rate, provide the motive force of the mobile phase. The sample components will migrate through the Rotor adsorbent as circular bands and will have differing affinities for the adsorbent versus the mobile phase. This

What are the advantages of centrifugal TLC vs. other separation methods?

- 2 4 grams maximum sample loading goes way beyond TLC plate capacity
- No scraping of bands necessary; bands elute off rotor
- 15 20 minute separation/elution times compared with 1 - 2 hour plate development time
- Reusable circular rotors; wash clean with Acetonitrile or Methanol
- Elution speed controlled by both solvent pump speed and RPM control

causes them to migrate at different rates affecting separation.

The circular bands eventually migrate to the outer edge of the adsorbent ring. The flowing mobile phase stream containing the component bands is ejected from the edge of the Rotor much like water from a rotating wet tire. A specially shaped collection channel around the outer perimeter of the Rotor collects the eluting mobile phase. The entire collection vessel is angled so that gravity drains the eluent to the exit port where it can be collected. The design of the vessel preserves the integrity of the sample bands so as to prevent remixing of the sample component bands.

Advantages

The CycloGraph centrifugal chromatography system combines the advantages of both preparative TLC and Column Chromatography. It delivers fast, efficient separations. Most separations occur in twenty minutes or less.

Fast separations are a result of the centrifugal action of the spinning Rotor driving the mobile phase through the adsorbent layer. The velocity of the mobile phase enables the use of smaller particle adsorbents. The Rotors for the CycloGraph use a 15 micron average particle adsorbent bed, similar to TLC. This smaller particle bed versus the 35-75 micron cut typical of low pressure column chromatography allows a higher degree of separation efficiency.

Centrifugal action combined with the use of a solvent pump to apply the mobile phase allow complete control of solvent velocity profile. Typical flow rates are in the range of approximately 2 - 3 ml/min per millimeter of adsorbent thickness. Tight bands mean eluted fractions can be collected in a minimum volume of solvent.

The solvent metering pump also makes the use of step gradients easy to perform with the CycloGraph. The inlet of the pump may be switched from a weak solvent to a more polar solvent during a run. This enables compounds with a wide range of polarities to be separated. Complex samples such as natural products are quickly separated into component classes.

Sweeping the polarity of the mobile phase from weak to strong makes it easy to reuse a Rotor for many sample separations. By concluding the run with a strong solvent, otherwise strongly adsorbed compounds will be flushed from the Rotor. This enables a Rotor to be reused for dozens of samples.

Separations on the CycloGraph scale easily and quickly. Rotors are available from one mm thick up to 8 mm thickness. For difficult cases, eluted components that are only partially resolved, can



be cycled back onto the spinning Rotor. This effectively allows the chromatographer to increase "the length of the column" without the associated disadvantages.

Separations on the CycloGraph can be monitored as they happen with the built in UV254 lamp. Separated substances which absorb 254 nm light will be visible against the fluorescent green background of the Rotor. Optionally a UV366 source may be used to detect intrinsically fluorescent components.

Chemical integrity of sensitive sample components can be preserved even with the use of active silica gel adsorbents. Unlike TLC where the sample solvent is evaporated before chromatography, the sample introduced on CycloGraph need never be exposed to dry adsorbent. This minimizes the possibility of reaction of sample components on the active adsorbent surface. Further control of the atmosphere is possible by flushing the vessel with nitrogen gas during chromatography.

CycloGraph[™] Rotors

ANALTECH'S Rotors are produced using clean, virgin glass blanks and high performance adsorbents. The glass is precision cut and thoroughly cleaned to provide unmatched balance and adsorbent adhesion.

Our high performance adsorbents have a narrow particle size distribution allowing minimum diffusion of separated bands while maximizing flow rates. Rotors have been formulated with the optimum amount of binder to produce a stable surface suitable for scraping without being soft, loose, or powdery. This formulation also helps extend the working life of the Rotor.

Due to stringent manufacturing controls during the coating and drying processes, rotors exhibit uniform, bubble-free layers. Therefore, bands remain concentric during the separation process

Features

- 1.Variable speed motor (100-1400 RPM)
- 2.Integrated 4-watt UV lamp for hands-free viewing of the sample
- 3.On/Off switches for the motor and UV lamp
- 4.Adjustable plane angle setting of vessel (0-30 degrees)
- 5.Switched solvent pump outlet
- 6.Ultra quiet & accurate low flow solvent pump (0-15ml/min)
- 7.Compatible with all common chromatography solvents including acetic acid

Included with the System (Catalog No. 87-40)

- CycloGraph[™] instrument
- · Solvent pump with necessary tubing
- Test Dye
- Instruction manual
- One Year Warranty

Optional Accessories

- Digital Speed Monitor
 (87-43)
- Rotor Dessicator/Storage Chamber (holds up to 10 Rotors) (50-05)

allowing collection of tight, narrow bands in a minimum elution volume.

Silica Gel GF Rotors are available in 2000um, 4000um, 6000um or 8000um nominal thicknesses (see price sheet). A uniformly distributed UV 254 nm fluorescent indicator is included in all rotor formulations to aid the user in following the course of separations.

A recent enhancement to these Rotors has made it even easier for chromatographers to use the CycloGraph. Previously Silica Gel Rotors needed to be scraped in the lab taking up valuable time while also making a mess in the process. Now, Rotors can be purchased pre-scraped and ready to use right out of the package. Not desiring to force this decision on all users, Analtech still sells the original un-scraped Rotors as well as bulk powder for those who choose to make their own.

The CycloGraph was designed for preparative separations on silica gel, however, other adsorbent layers are still under development.



CycloGraph Instrument

Dimensions:	340 (h) x 260 (d) x 298 (w) mm
Weight:	7.5 kg (16.5 lbs)
Chassis:	stainless steel (304), polycarbonate

Liquid Contacting Parts

Vessel:	polyformaldehyde resin (Delrin)
Vessel Lid:	Teflon
Wick:	Polyester
Tubing:	2mm Teflon
Tubing Filter:	Polyester
Rotational Speed:	720* rpm at 60 hz (as shipped) 100-1440* rpm at 50 hz (variable) 100-1770* rpm at 60 hz (variable)
Rotational Speed: Vessel Pivot:	720* rpm at 60 hz (as shipped) 100-1440* rpm at 50 hz (variable) 100-1770* rpm at 60 hz (variable) 0° (horizontal) to 30°
Rotational Speed: Vessel Pivot: Gas Inlet Port:	720* rpm at 60 hz (as shipped) 100-1440* rpm at 50 hz (variable) 100-1770* rpm at 60 hz (variable) 0° (horizontal) to 30° 1/4" hose barb
Rotational Speed: Vessel Pivot: Gas Inlet Port: Tubing Port:	720* rpm at 60 hz (as shipped) 100-1440* rpm at 50 hz (variable) 100-1770* rpm at 60 hz (variable) 0° (horizontal) to 30° 1/4" hose barb 1/4-28

* speeds listed are approximate

UV Lamp:4 watt, 254 nm (366 nm optional)
(with ON/OFF switch)Lamp Dimensions:24 (h) x 36 (d) x 146 (w) mmMotor:100-120 VAC, 50/60 hz, 0.7 amps
(with ON/OFF switch)Outlet:100-120 VAC, 50/60 hz
(with ON/OFF switch)Fuse:3 ampsRotors Accepted:1 to 8 mm thickness, 9.5" diameter

Solvent Pump

Outer Dimension:	127 (h) x 127 (d) x 102 (w) mm
Material:	ceramic or stainless steel and carbon
	(contacting part to liquid)
Weight:	1.1 kg (2.4 lbs)
Flow Rate:	0-15 mL/min
Electricity:	110 VAC, 60 hz, .08 amps or
	220 VAC, 50 hz, .04 amps

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