Representative Separation Projects at Government Labs

National Science Foundation
Chemical and Biological Separations (CBS)
http://www.nsf.gov/funding/pgm_summ.jsp?pims_id=13363

The Chemical and Biological Separations (CBS) program supports fundamental research on novel methods and materials for separation processes. These processes are central to the chemical, biochemical, materials, energy, and pharmaceutical industries. A fundamental understanding of the interfacial, transport, and thermodynamic behavior of multiphase chemical systems as well as quantitative descriptions of processing characteristics in the process-oriented industries is critical for efficient resource management and effective environmental protection. The program encourages proposals that address emerging research areas and technologies, have a high degree of interdisciplinary thought coupled with knowledge creation, and integrate education and research.

Areas of emphasis include separation of biological molecules and separations that lead to environmentally benign processing. Much of the current work involves the investigation of new membrane materials such as novel polymers, zeolites, mixed matrix materials, carbon nanotubes, biological and biomemetic materials, and glasses.

Projects on modeling transport processes - - especially at the molecular level - - in membranes are supported by the program. For the hydrogen economy, membranes that selectively transport atomic, molecular, or ionic hydrogen and oxygen are required.

Current membrane materials often lack sufficient selectivity to eliminate critical contaminants from the hydrogen stream. Membrane materials to prevent fuel crossover in fuel cells are also being sought. Adsorption on materials ranging from hydrogels to micas is being studied. New processes for drinking water purification rely on the development of new materials for membranes and adsorbents, which are supported by CBS.

Research topics in CBS include fundamental molecular-level work on:

- Biochemical separations and purification processes
- Microporous and novel molecular-recognition adsorbents
- Self-assembly in the synthesis of adsorbents and membranes
- Nanostructured materials for separations
- Fuel-cell membranes
- Biomimetic materials for separations
- Chiral separations
- Separations for environmentally benign processing
- Novel polymeric and ceramic membranes
- Hybrid separation processes
- Control and separation of organic crystalline materials
- Separations using ionic liquids
- Purification of drinking water

- Membranes for ion-selective sensors
- Adsorption and chromatography
- Field (flow, magnetic, electrical) induced separations.
- Separation of molecular constituents from blood
- Thermodynamics and transport simulations for the design of separation processes
- Combinatorial design of separation systems
- Rational ligand design for separations

National Institute of Standards and Technology http://www.nist.gov

Separation technologies play a hidden but vital role in manufacturing. Users of specialty-separation and high-volume separation methods together represent approximately $1.2 trillion in product shipments. Separations currently constitute up to 90 percent of the processing costs in the biotechnology industry and up to 70 percent of capital and operating costs in high-volume chemical applications.

Advanced Technology Program: http://www.atp.nist.gov/
ATP helps industry invest in longer-term, high risk research with payoffs far beyond private profit. One of the 40 some technology areas supported by the ATP is separation technology. Examples of supported projects that involve strong focus on membrane technology are:

- Development of Robust Fuel Flexible Desulfurization Systems for Clean Power Solutions
  http://jazz.nist.gov/atpcf/prjbriefs/prjbrief.cfm?ProjectNumber=00-00-7436
- Ultraslow Methanol Crossover Membranes for Higher Energy Density Direct Methanol Fuel Cells
  http://jazz.nist.gov/atpcf/prjbriefs/prjbrief.cfm?ProjectNumber=00-00-7744

Environmental Test Program

Solid/Gas Separation
Design and characterization of quartz fiber filters to produce an air particulate matter filter Standard Reference Material.
http://www.cstl.nist.gov/div837/Division/techac/1999/PM2_5UrbanParticulate.htm

Sampling and measurement of atmospheric aerosol carbon from experimental forest fires using quartz-fiber filters backed by an additional filter to account for adsorbed gas-phase compounds and particle losses.
http://www.cstl.nist.gov/div837/Division/techac/2000/CarbonFire.htm

Separation and quantification of fossil- (petroleum derived) or biomass- derived carbon particulate matter http://www.cstl.nist.gov/div837/Division/techac/0_Pre99/NFRAQS.htm

Liquid/liquid Separation
Projects include fluid-based physical processes and systems, *including separations*, low-temperature refrigeration, and low-temperature heat transfer and flow.

Nano and ultra filtration

- Yoon, Y., Amy, G., Cho, J., and Pellegrino, J., *"Systematic Bench-scale Assessment of Perchlorate (ClO_4^-) Rejection Mechanisms by Nanofiltration and Ultrafiltration Membranes,"* Separation Sci. Tech., 39, 2105-2135 (2004).
- Lattice Boltzmann, "Model of Fluid Phase Separation" http://ciks.cbt.nist.gov/~garbocz/lbseparate/mft4.html
- Microfluidic separation and measurement of biomolecules http://www.cstl.nist.gov/projects/fy06/health0683105.pdf

Solid/liquid separation
Sorting and manipulating nanotubes by length and type using size exclusion chromatography, NIST Polymer Lab, Gaithersburg, MD
(http://www.ncnr.nist.gov/programs/sans/pdf/publications/0526.pdf)

Department Of Energy

http://www.ne.doe.gov/pdfFiles/afciFy2005StatusRptToCongress.pdf

Separation Technology Development
The Advanced Fuel Cycle Initiative program is investigating technologies in two primary separations areas–advanced aqueous-based processing and pyroprocessing. Many aqueous-based approaches to treat spent nuclear fuel exist. The Uranium Extraction Plus (UREX+) method is an advanced aqueous process with significant potential for meeting proliferation-resistant separations objectives while minimizing the waste generation historically associated with aqueous separations technologies. While UREX+ has great potential to address the spent fuel challenge associated with today's light water reactors, pyroprocessing is potentially best suited to address the needs of next generation fast neutron spectrum reactor fuels.

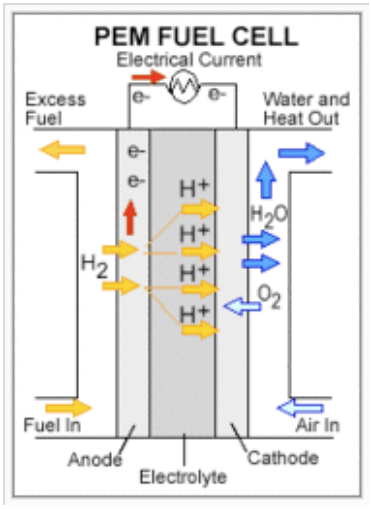
Advanced Aqueous Processing - UREX+ Experiment
Experiments completed in FY 2003 as well as continued testing in FY 2004 have proven the advanced, aqueous-based UREX+ technology to be capable of removing uranium from spent fuel at such a high level of purity that the uranium is expected to be sufficiently free of high-level radioactive contaminants to allow it to be disposed of as low-level waste or reused as reactor fuel. These laboratory-scale tests have proven uranium separation at purity levels of 99.999 percent. If spent fuel were processed in this manner, the potential exists to reduce significantly the volume of high-level waste requiring disposal in a geologic repository.

Pyroprocessing Development
Pyroprocessing technology development activities since FY 2003 have focused on treating highly-enriched, sodium-bonded driver fuel while investigating alternatives to more cost-effective technologies for processing sodium-bonded blanket fuel. This focus is reflected in the Report on the Preferred Treatment Plan for EBR-II Sodium-Bonded Spent Nuclear Fuel (June 2003).

Argonne National Laboratory

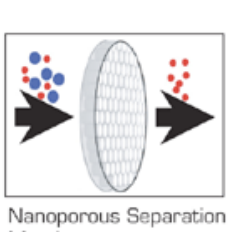
http://www.cmt.anl.gov

Argonne National Laboratory occupies 1,500 wooded acres in DuPage County, Ill, about 25 miles southwest of Chicago.

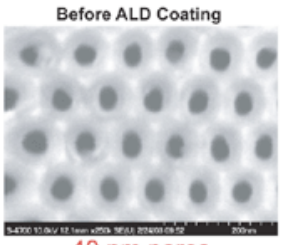


Nanoporous Catalytic Membranes

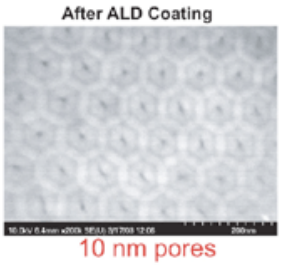
Conversion of the feedstocks into chemicals, diesel fuel or gasoline using catalytic membranes would be a great step towards energy independence. Existing catalysts for this process provide low selectivity over the hydrocarbon product distribution, and make inefficient use of the precious metal catalysts. Argonne aims to fabricate novel nanostructured catalytic membranes that will overcome these limitations and enable the cheap and efficient synthesis of hydrocarbons for gasoline and diesel fuel. Argonne's ultra-uniform nanoporous catalytic membranes are based on anodic aluminum oxide (AAO). By using atomic layer deposition (ALD) thin film growth techniques AAO pore size is optimized for reactant/catalyst contact time as well as to provide filtration capability.



Nanoporous Separation Membrane



40 nm pores



10 nm pores

National Renewable Energy Laboratory, Golden, CO

http://www.nrel.gov/

Catalytic Membranes (Industrial Technology Program)

The NREL is partnering with several companies to investigate catalytic reaction/separation membrane as a method of reducing production costs. Various industrial processes, from pharmaceutical production to polymer formation, involve homogeneously catalyzed organic reversible reactions. A co-product of many of these reactions is water, and its removal allows the equilibrium reaction to shift towards the greater formation of desired product. Its removal also results in faster reaction and purer products, which thereby reduces production costs.

http://www1.eere.energy.gov/industry/chemicals/pdfs/compact_membrane_sys.pdf

Polymer Electrolyte Membrane (PEM) Fuel Cells

Hydrogen, Fuel Cells and Infrastructure Technologies Program

The function of the PEM is the separation of charges within the cell. Positive ions are conducted through the membrane where as negative ions are excluded. Of the approximate 10 type of fuel cells that have been or are being developed, PEM fuel cells will have applications in the following areas:

- Portable PEM-based fuel cell systems with power ratings of less than 1 kilowatt
- Portable PEM fuel cells include hydrogen-fueled, direct-methanol-fueled, and reformed-methanol-fueled PEM fuel cells.
- Backup power and remote power PEM fuel-cell systems with power ratings of 1 kW to 5 kW. Hydrogen is the fuel for these applications.
- Residential power PEM fuel-cell systems with power ratings of 1 kW to 10 kW. These systems typically operate with an on-site reformer. The choice of fuels includes methanol, methane (natural gas), and propane.
- Stationary power PEM fuel-cell systems operating at power ranges from 50 kW to 250 kW. These systems operate with an on-site reformer. Methane is the typical fuel for these systems.
- Industrial vehicles powered by 10 kW to 50 kW PEM fuel cells. These vehicles include forklifts and people movers. Hydrogen is the fuel of choice for these applications.
- Automotive PEM fuel cells with PEM systems power ratings from 50 kW to 100 kW. Hydrogen is the fuel of choice for the light-vehicle applications.

- PEM systems with power ratings from 100 kW to 300 kW for heavy-duty vehicles such as buses. Hydrogen is the fuel of choice for these applications.

National Energy Technology Lab www.netl.doe.gov

Ceramic Membranes
The water-gas-shift (WGS) reaction, $CO + H_2O \leftrightarrow H_2 + CO_2$, is used to increase the hydrogen content of synthesis gas. One approach for overcoming this limitation is to carry out the reaction in a reactor with walls that are CO_2 permeable. This continuously removal of CO_2 from the system should allow the reaction to continue.
http://www.netl.doe.gov/publications/factsheets/project/Proj195.pdf

Ionic Liquid & Polymer Soluble membranes
Improvements in capture/separation have the greatest potential to affect the cost of CO_2 mitigation, and membrane technology holds significant promise in this area. One current research focus is on a class of salts known as ionic liquids. Certain ionic liquids have high solubility for CO_2 compared to H_2 and other light gases, greater diffusivities than polymers, and stabilities to temperatures above 200 °C. These characteristics lead to the conclusion that ionic liquids have the potential to form the basis for a new and superior class of CO_2 selective membranes. Another area of focus is on polymers which dissolve readily in CO_2. These polymers have high molecular affinity for CO_2 and are promising candidates for membrane materials.
http://www.netl.doe.gov/publications/factsheets/rd/R&D047.pdf

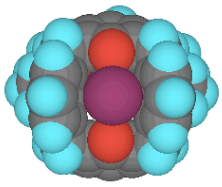
Oak Ridge National Laboratory http://www.ornl.gov

The Chemical Separation's Group Capabilities
- Chemical separations
- Analytical chemistry
- Chemical analysis
- Solvent extraction
- Ion exchange
- Hydrometallurgy
- Radiochemistry
- Design and synthesis of novel ligands and extractants
- Thermodynamic characterization of extraction behavior and underlying equilibria.
- Structural analysis of ligands and complexes by X-ray, NMR, and spectroscopic methods
- Fundamental research to process development

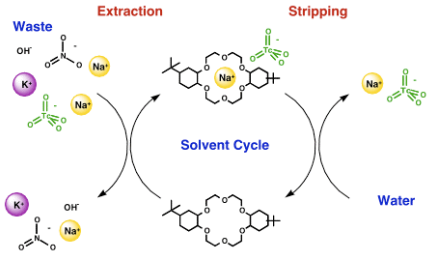
Emphasis Areas
- Crown ethers, macrocycles, and designer ligands for recognition and transport of cations and anions
- Novel materials and processes for waste remediation, environmental restoration, and decontamination
- Metal-organic frameworks
- Metallo-organic compounds for sensors, catalysis, and other applications

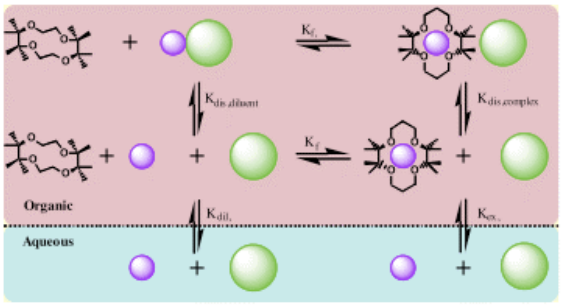
Accomplishments

- Separating Radioactive ^{137}Cesium - development of a new solvent extraction process for separating radioactive ^{137}cesium from the nation's troublesome high-level wastes. (TechTransfer to IBC Advanced Technologies)
- Separation for Removal of Radioactive Technetium. Technetium is a long-lived radioactive fission product found in wastes stored at several DOE sites. Research in the Chemical Separations Group of the Chemical and Analytical Science Division at ORNL has recently suggested extractive methods using Crown ether complexing with Na^+ and K^+ ions. This process is described in a patent and publications from the 1990's.



Extraordinary selectivity for Cs^+ ion vs. Na^+ ion is > 10^4, due to the excellent match of the size of the calixarene cavity and the ionic diameter of Cs^+. Picture shows end-on view with foreground atoms deleted.





Removal of Trace Amounts of Radioactive Technetium from Groundwater

Researchers at Oak Ridge created the bifunctional resin they call "BiQuat, that has both small and large positively charged groups within the resin. The small groups promote fast exchange, while the large groups provide highly selective sites. In field tests at Paducah, BiQuat performed five-fold better than the resin used at the site.

TechTransfer → material in commercial development by Purolite Company

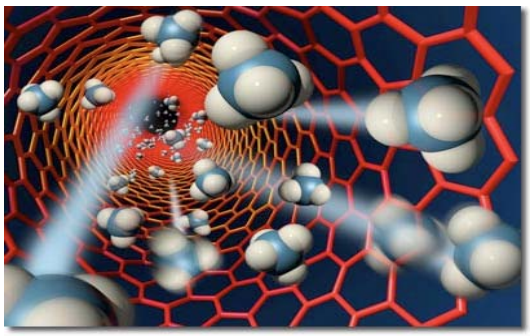
TechTransfer → radioanalytical techniques to East Tennessee Radiometric Analytical Chemicals, Inc.

Increased Selectivity and Separation Capability for Alkali Metal Cations

Both phenolic and calixarene-crown extractants possess the same ability to bond to alkali metal cations through the pi-cloud of a benzene ring. In the case of calixarene-crowns, the pi-bonding takes place in a pre-organized molecular cavity, whereas simple 2-benzylphenols apparently self-assemble about the cation. This concept has implications for nuclear waste clean-up.

Lawrence Livermore National Laboratory

https://www.llnl.gov/



Scott Dougherty, LLNL

Desalination
The LLNL has created a membrane made of carbon nanotubes and silicon that may offer less expensive desalination. The nanotubes, special molecules made of carbon atoms in a unique arrangement, act as the pores in the membrane. The pores are so small that only six water molecules could fit across their diameter. The super smooth inside of the nanotubes allow liquids and gases to rapidly flow through, while the tiny pore size can block larger molecules.

Laser isotope separation
It is very difficult to separate a desired isotope of a chemical element from the remaining isotopes for uses ranging from medicine to energy to weapons applications. Over the past 2 decades scientists at the LLNL have developed a technique that is based on the fact that different isotopes of the same element, while chemically identical, absorb different colors of laser light. Therefore, a laser can be precisely tuned to ionize only atoms of the desired isotope, which are then drawn to electrically charged collector plates.

National Science Foundation www.nsf.gov/

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Areas of emphasis include separation of biological molecules and separations that lead to environmentally benign processing. Much of the current work involves the investigation of new membrane materials such as novel polymers, zeolites, mixed matrix materials, carbon nanotubes, biological and biomemetic materials, and glasses. Projects on modeling transport processes - - especially at the molecular level - - in membranes are supported by the program. For the hydrogen economy, membranes that selectively transport atomic, molecular, or ionic hydrogen and oxygen are required. Current membrane materials often lack sufficient selectivity to eliminate critical contaminants from the hydrogen stream. Membrane materials to prevent fuel crossover in fuel cells are also being sought. Adsorption on materials ranging from hydrogels to micas is being studied. CBS also supports new processes for drinking water purification rely on the development of new materials for membranes and adsorbents,

Department of the Interior, Bureau of Reclamation
http://www.usbr.gov/

The Desalination and Water Purification Research & Development (DWPR) Program
http://www.usbr.gov/pmts/water/newsletter/Water/2001sum.html
Through the Desalination & Water Purification Research & Development (DWPR) Program, Reclamation has formed partnerships with private industry, universities, local

communities, and others to address a broad range of desalting and water purification needs. The overall program objective is to reduce the cost of desalting and water purification technologies in order to augment U.S. water supplies. The DWPR program focuses on:

- Research and studies on desalination technologies and related issues that pushes the state of the art forward so costs can be reduced.
- Development and demonstration activities to test technological advancements, confirm economics, and gain public acceptance.

NASA National Space Science and Technology Center, Huntsville, AL
http://www.nsstc.org/

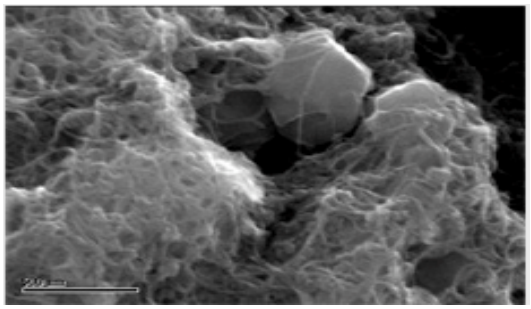
Gas Separation
SBIR Interest Area: The continuing rising costs of helium justifies the construction of an economic system that will separate helium gas from gas streams that would normally be considered waste gas. Since almost all of NASA's launch and other propellant related test operations use helium and hydrogen, this system would be capable of recovering these valuable gases to the point that they could be separated and recycled.

Ames Research Center, Moffett Federal Airfield
http://www.nasa.gov/centers/ames/home/index.html

Gas Separation
The Adsorption Laboratory in the Bioengineering Branch at NASA Ames Research Center is equipped to successfully handle development activities related to gas separation, purification, storage, and compression processes of gases. ARC has extensive computer, laboratory, manufacturing, electronics labs, materials processing labs, and test facilities to support the design, development, testing and certification of In-Situ Resource Utilization components, systems, and experiments.

Carbon Nanotube Membranes or “Buckypaper”
http://www.nasa.gov/centers/ames/research/technology-onepagers/nanoscale_prt.htm

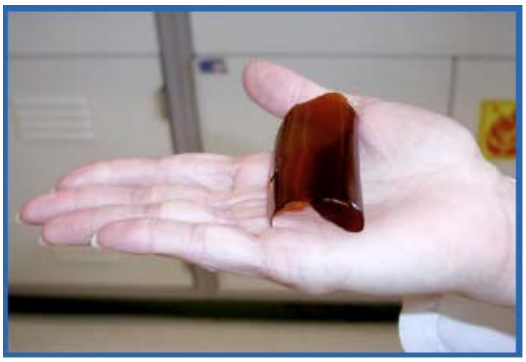
Nanoporous media may have practical applications in separations and catalysis, allowing for size-selective molecular separations or high surface area catalysis. Applications in this general vein include air purification for manned space missions. Micro- and nano-electromechanical systems are being developed for a variety of applications including gas sensing and materials analysis.



Glenn Research Center
http://technology.grc.nasa.gov/tops/TOP300178.pdf

Organically Modified Silicates Polymer Membranes (ORMOSIL)
NASA Glenn Research Center has developed a series of ORMOSILs with acceptable ionic conductivity as well as thermal and dimensional stability that have applications as a proton-conducting membrane for PEM fuel cell applications, gas separation membranes properties, and solid electrolytes for lithium batteries.

Benefits:
- Simple fabrication
- Dimensionally stable, flexible films
- Good ion conductivity over a wide range of temperatures
- High pressure not required



USDA ARS, (CA)
http://www.ars.usda.gov/is/pr/2007/070626.htm

Membrane Separation of Ethanol
Ethanol to fuel cars, trucks and other vehicles might tomorrow take less energy to produce, thanks to a device invented by Agricultural Research Service (ARS) scientists in California. The technology will help to address the serious concern regarding the energy efficiency of bioethanol production. The researchers' invention, called a spiral-wound liquid membrane module, could potentially replace the widely used process of

distilling ethanol from fermentation broths. The module offers ethanol producers the important advantage of combining two separation processes, extraction and membrane permeation, in one piece of equipment.

Removal and Separation of Bran, Protein and Starch from Rice
http://www.ars.usda.gov/is/AR/archive/feb05/grain0205.pdf

For nearly 60 years, the processing of this starch has hardly changed, relying always on the action of a corrosive alkali, sodium hydroxide, to slowly dissolve rice protein and release of starch. Typically, rice is steeped in sodium hydroxide for several hours to dissolve the protein and let the starch molecules break free. But that degrades the protein, leaving it bitter-tasting and unfit for human consumption. The new approach instead relies on very high pressure, supplied by a special homogenizer known as a microfluidizer, to physically split apart the starch/protein agglomerates. The starch and protein components can then be separated by traditional density-based separation processes.

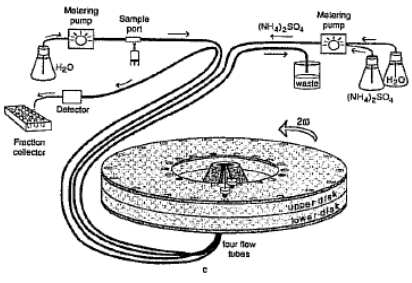
Other Research Areas Dealing With Separation Technology

- Separation protein & starch
- Swine waste treatment/NH4 Separation & Removal
- Separation and identification of lipid structures
- Separation of males and female sperm in animals and humans

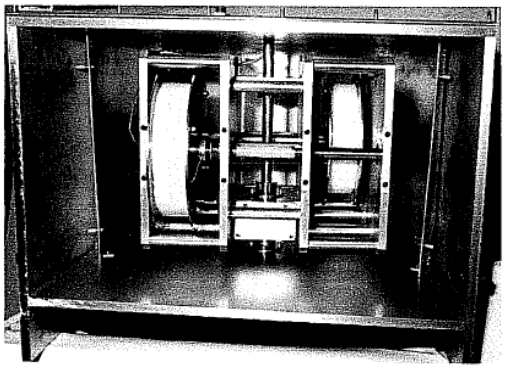
National Institute of Health, National Cancer Institute
http://ccr.cancer.gov/staff/staff.asp?profileid=5657
Single Cell Separation - “Flow Cytometry in Immunobiology”
Flow cytometry (FCM) is a unique experimental technology that provides rapid, quantitative, multiparametric, single cell analysis and separation. The FCM lab has two customized three-laser (two tunable argon or krypton and rhodamine 6G argon-pumped dye laser) flow cytometers with electronic cell separation capabilities that are operated by staff of the Flow Cytometry Laboratory in support of multiple research projects. These investigations involve quantitative, single cell analyses of parameters associated with cells freshly prepared from different species/tissues as well as a wide spectrum of in vitro cultured cells. Cell-associated molecules are measured with a variety of probes, most often fluorochrome-labeled monoclonal antibodies. The laboratory specializes in multicolor immunofluorescence analysis (up to six colors), rare event analysis, and cell separation.

Chromatographic Separation Apparatus and Method
U.S. Patent No. 6,379,973
This technology was exclusively licensed to CC Biotech, a small Maryland-based, biotechnology company formed specifically to develop, manufacture and sell countercurrent chromatographic devices to the research community and the pharmaceutical industry.

This invention pertains to an apparatus and method for separating bio-molecules using a salt gradient (e.g., ammonium sulfate) applied in a spiral flow channel divided by a dialysis membrane. The channels are compartmentalized into upper and lower rotary discs spinning anti-clockwise to each other. A sample containing compounds to be purified is introduced in a buffered solution into the lower channel flows in one direction while the salt flows in the other. The concentration of salt permeates the membrane and precipitates the large molecules on the other side. For example, proteins or polymers are precipitated sequentially in the channel and centrifugal forces force the precipitate to the outer rim as it moves along the liquid stream.



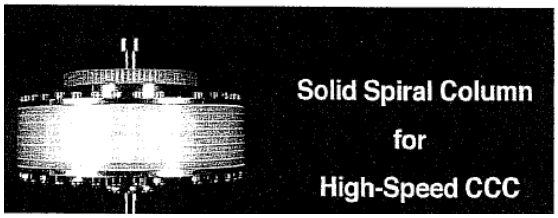
U.S. Patent Application "Variable-Position Cross-Axis Synchronous Coil Planet Centrifuge for Countercurrent Chromatography,"



This device is a cross-axis synchronous flow-through coil planet centrifuge which provides changeability in the position of the coils relative to the axis of rotation of the centrifuge. The advantage of this feature is to allow adjustment of the centrifugal force operating on the coils to accommodate different types of separations. The coils are arranged in columns mounted to column holders that in turn can be engaged to the rotary frame of the centrifuge in positions in which the column holders intersect and do not intersect the rotary frame axis. This arrangement allows for larger coils which can hold between 200 ml and 800 ml of sample.

U.S. Patent Application "Method and Apparatus for Countercurrent Chromatography;"

This device and method is an improvement of the countercurrent chromatography device disclosed This patent pending device is a new spiral design capable of holding heavier solvent systems suitable for peptides and proteins (e.g., for natural products).



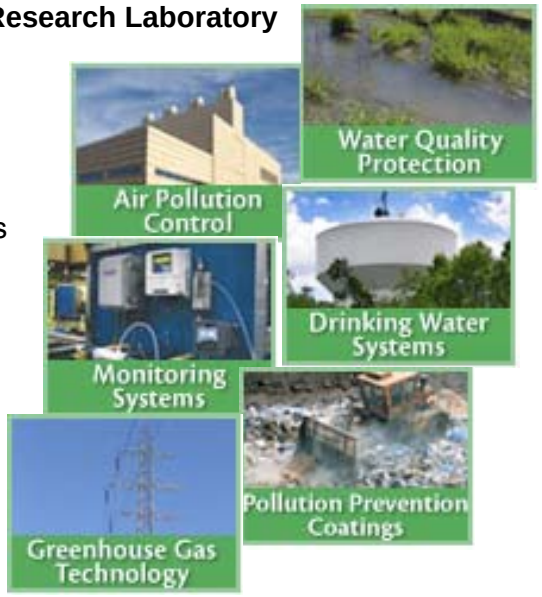
Naval Research Laboratory

http://www.nrl.navy.mil/

Separation of Infrared Spectra
(http://lep.gsfc.nasa.gov/code693/tdw03/proceedings/docs/session_5/Sternberg.pdf)
Infrared filters play an integral role on every infrared astronomy airborne and/or space mission. They isolate the desired infrared signal from more energetic short wavelength radiation, allow color temperature measurements, provide order sorting for grating spectrometers, and improve the signal-to-noise for Fourier transform spectrometers. The need for such devices is critical now, as space astronomy in particular begins to emphasize far infrared research programs in the new millennium.

Carbon Nanotubes
Scientists at the Naval Research Laboratory have successfully produced carbon nanotubes (CNTs) in high yields in bulk solid compositions using commercially available aromatic containing resins. The concentration of multi-walled carbon nanotubes (MWNTs) and metal nanoparticles can be easily varied within the shaped carbonaceous solid. Carbon nanotube containing fibers and films have also been formulated from the precursor compositions. The potential range of applications is huge, including structure, energy, sensors, separation/filtration, battery, electronic displays, and nanoelectronic devices.

Environmental Protection Agency: National Exposure Research Laboratory



Water Quality Protection Center Verified Technologies

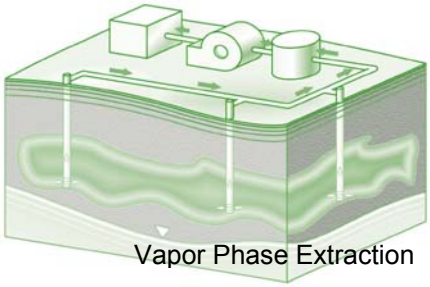
- Animal waste treatment - solids separation
- Chemically enhanced high-rate solids separation
- Decentralized wastewater treatment technologies
- Decontamination wastewater treatment technologies
- In-drain treatment technologies
- Mercury amalgam separation
- Residential nutrient reduction
- Storm water source-area treatment devices
- Ultraviolet (UV) disinfection
- Secondary effluent/wastewater reuse
- Vortex high-rate solids separation

Separation & Removal of VOC's From Water
The EPA's National Risk Management Research Laboratory (NRMRL) has maintained an active in-house pervaporation research program for over eight years. Early work dealt with the removal of multiple volatile organic compounds (VOCs) from water using existing and novel membrane materials. Today, the EPA continues to develop and evaluate new membranes for pervaporation of organic compounds that aid in recovery and reuse of those compounds. The focus of this work has been on membranes with superior separation properties and resistance to extreme environments, such as ceramic-supported polymeric membranes.

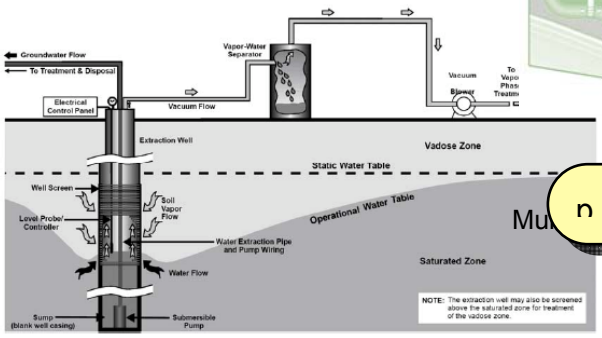
Separation & Removal of VOC from Soil

Soil vapor extraction systems generally have the following set-up. Once the gas is removed, the question is what to do with it. Depending on the properties of the vapor, the following processes can be utilized:

- Thermal
- Adsorption
- Biofiltration
- Emerging Technologies
  - -Non-thermal plasma
  - -Photolytic and photocatalytic technologies
  - -Membrane separation



Vapor Phase Extraction



Mu n Pervaporation Process