

New Chromatography Columns and Accessories at the 2004 Pittcon Conference, Part II

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This month's "Column Watch" is the conclusion of a two-part series in which Ron Majors examines the trends in column introductions at Pittcon 2004. In Part II, he discusses gas chromatography columns, sample preparation products, hardware, accessories and kits for chromatography and sample preparation.

The purpose of this report is to provide information about many of the new separation consumables and accessory products that were displayed at Pittcon 2004. In some instances, products that were introduced during 2003 but after Pittcon 2003 could be included for reasons of completeness. The information is based upon manufacturers' responses to a questionnaire mailed in December 2003. Because of space limitations and the fact that some of the manufacturers did not respond to the questionnaire, this report cannot be considered an exhaustive listing of all new products that were introduced in Chicago.

As in previous years, columns and other products recommended by their manufacturers primarily for biomolecule separations or sample preparation are denoted in the tables with the designation *BIO*. Some of these products can be used for general high performance liquid chromatography (HPLC) separations as well, but their main emphasis is on biological samples. I cite specific information about bioapplications where appropriate.

In part I, I described new columns for high performance liquid, reversed-phase, normal- and bonded-phase, ion-exchange- and ion, size-exclusion, and speciality chromatography.¹ This month, I will look at gas chromatography (GC) columns, sample preparation products, hardware, accessories and kits for chromatography and sample preparation.

GC and Supercritical Fluid Chromatography Columns

Eight companies contributed new GC column information for Pittcon 2004, and one company introduced a series of columns for analytical and preparative supercritical fluid chromatography SFC (Table 1). In recent years, there has been a trend in the introduction of ultralow-bleed stationary phases that allow better detection limits and less baseline drift during temperature-programmed separations. This year, three companies, Macherey-Nagel, Varian and VICI introduced new capillary columns with greatly improved bleed specifications compared with previous versions. Capillary columns with inner diameters ranging from 0.10 mm (small bore) to 0.53 mm (large bore or megabore) were the most popular sizes. This year, most introductions were wall-coated open tubular (WCOT) columns with two new porous-layer open-tubular (PLOT) columns and one micropacked column for SFC.

Many of the general-purpose GC capillary columns displayed at Pittcon 2004 were crosslinked alternatives to popular coated phases. Crosslinked, chemically bonded or immobilized phases are more stable, have lower bleed and can usually operate at higher temperatures than coated phases. Another advantage of the bonded phases is their ability to be solvent-rinsed. If a column becomes contaminated by high-boiling-point compounds, inorganic compounds or other non-volatile

impurities that can cause drifting baselines, bleed and other undesirable occurrences, the column can be disconnected, washed (from outlet to inlet) with various solvents, and placed back into the gas chromatograph for further use.

Application-specific columns are always popular introductions at the exposition. Columns for permanent gases, light hydrocarbons, simulated distillation, drug testing, chiral compounds, environmental samples, flavours and fragrances, and *cis-trans* fatty acid methyl esters were among those submitted. Such columns are usually tested with the compounds to be assayed and test chromatograms shipped with the column.

NanoTek displayed a unique column module. The packed capillary column is produced on a nanofabricated titanium-aluminum alloy module with a built-in temperature controller. The entire module is small and weighs only 3–7 oz depending upon the configuration. It replaces the column-oven compartment of a conventional GC instrument and is only 1/1000 of the size of a standard GC oven. It is used for portable GCs and other monitoring applications.

Sample Preparation Products

As indicated in Table 2, Pittcon 2004 was another productive year for the sample preparation arena, with 26 new products or families of products displayed. A majority of these products fits into the

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solid-phase extraction (SPE) category with formats ranging from the traditional cartridges (a majority) and discs to 96-well plate configurations.

During the past several years, the 96-well SPE and filter-plate design has proliferated. This year, the number of 96-well plates actually exceeded the number of traditional cartridge and disc introductions. The predominant driving force for the plate configuration has been the high-throughput requirements placed on the sample preparation–chromatography–mass spectrometry (MS) laboratories by the pharmaceutical industry for drug screens and combinatorial chemistry needs. As sample loads increase to hundreds per day, the need for rapid turnaround of analytical results dictates automation, and the robotics and liquid-handling systems have geared up to meet the demand. For SPE plates, a small amount of solid-phase sorbent — generally 10–100 mg — is placed into each well and the packing is sandwiched by frits to form a minibed. Then, liquids for conditioning, loading sample, rinsing and eluting are added by liquid-handling workstations.

The 96-well filter plates, sometimes referred to as crash plates, are used for the automated filtration of large numbers of samples. Each well of a filter plate has a membrane filter. The filter plates are used most often in the measurement of drugs and drug metabolites in blood plasma.

Users, usually via automation, first add acetonitrile (sometimes containing a small amount of trifluoroacetic acid) to each well in the plate, and then add a small volume, usually a few hundred microlitres, into each well with mixing. The protein in the plasma precipitates (crashes), and the supernatant is filtered into a collection plate for further processing. Because in this process there are no selective interactions and all non-precipitated compounds remain in the supernatant, the extract might not be as clean compared with SPE. However, a reduced amount of method development time is required. If LC–MS or LC–MS–MS is used, after protein precipitation and filtration, the extract cleanliness may be sufficient.

Alternative devices also suited to automation are SPE pipette tips. In this design, a small amount of packing material is either held in the tip by frits, inserted as a plug of monolith packing or coated on the inside of the tip to allow easier passage of fluid. This design has proven useful for desalting small amounts of protein and other biological samples for study by LC–MS. Several manufacturers introduced pipette tips with clean-up capability. Examples of new SPE pipette tips and a deep-well 96-well plate are shown in Figure 1.

In proteomics studies, tiny amounts of protein-containing samples from cells and other biological forms are investigated in attempts to relate minor up-regulated or down-regulated proteins to disease states

Companies Listed in This Column

- 3M Bioanalytical, St. Paul, Minnesota, USA
- Agilent Technologies Inc., Wilmington, Delaware, USA
- Alltech Associates Inc., Deerfield, Illinois, USA
- Analytical Sales and Services, Mahwah, New Jersey, USA
- Argonaut Technologies Inc., Foster City, California, USA
- Chromatography Research Supplies Inc., Louisville, Kentucky, USA
- Dionex Corp., Sunnyvale, California, USA
- Dychrom, San Jose, California, USA
- Glygen, Columbia, Maryland, USA
- Horizon Technology Inc., Atkinson, New Hampshire, USA
- Kimble/Kontes, Vineland, New Jersey, USA
- Macherey-Nagel GmbH & Co. KG, Düren, Germany
- Merck KGaA, Darmstadt, Germany
- MicroSolv Technology Corp., Long Branch, New Jersey, USA
- Millipore Corp., Billerica, Massachusetts, USA
- Mo Bio Laboratories, Carlsbad, California, USA
- NanoTek, Tucson, Arizona, USA
- New Objective Inc., Woburn, Massachusetts, USA
- Phenomenex, Torrance, California, USA
- Pickering Laboratories Inc., Mountain View, California, USA
- Polymer Laboratories Ltd, Church Stretton, Shropshire, UK
- Porvair plc, Norfolk, UK
- Princeton Chromatography, Princeton, New Jersey, USA
- Quadrex Corp., Woodbridge, Connecticut, USA
- Rheodyne LLC, Rohnert Park, California, USA
- Restek Corp., Bellefonte, Pennsylvania, USA
- Scivex (formerly Upchurch Scientific), Oak Harbor, Washington, USA
- SGE Inc., Austin, Texas, USA
- Sigma-Aldrich, St. Louis, Missouri, USA
- Spark Holland BV, Emmen, The Netherlands
- Sun SRI, Wilmington, North Carolina, USA
- Supelco, Bellefonte, Pennsylvania, USA
- Swagelok Co., Solon, Ohio, USA
- Target Discovery, Palo Alto, California, USA
- United Chemical Technologies, Bristol, Pennsylvania, USA
- Varian Inc., Palo Alto, California, USA
- Varian Middelburg, Middelburg, The Netherlands
- VICI Gig Harbor Group, Gig Harbor, Washington, USA
- Waters Corp., Milford, Massachusetts, USA

Figure 1: New SPE devices: (a) OMIX (Varian); (b) NuTip (Glygen) SPE pipette tips; and (c) a deep-well 96-well SPE plate (3M).

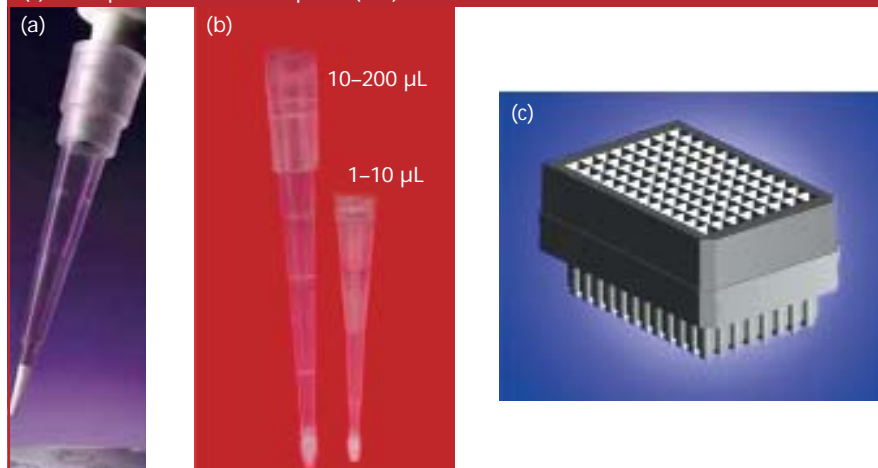


Table 1: GC and SFC columns at Pittcon 2004.

Product Name	Supplier	Type	Stationary Phase	Film Thickness (μm)	Inner Diameter (mm)
Alumina PLOT	Supelco	PLOT	Alumina	5, 10	0.32, 0.53
ColOvn-P	NanoTek	Packed capillary	Various	Various	Various
DB-WaxFF	Agilent Technologies	WCOT	88% cyanopropyl-dimethylpoly-siloxane	0.2	0.1
EZ-Guard FactorFour	Varian	WCOT	Various	0.1–1.0	0.25
FactorFour VT-200ms	Varian	WCOT	Trifluoropropylmethylpoly-dimethylsiloxane	0.1–1.0	0.25, 0.32, 0.53
FactorFour VT-35ms	Varian	WCOT	35% phenyldimethylpolysiloxane	0.1–1.0	0.25, 0.32, 0.53
High Temperature Simulated Distillation	VICI Gig Harbor	WCOT	Methyl polysiloxane	0.9–1.5	0.53
HP-88	Agilent Technologies	WCOT	88% cyanopropyl-dimethylpoly-siloxane	0.2	Popular
Hydrodex beta-TBDAC	Macherey-Nagel	WCOT	Heptakis-(2,3-di- <i>O</i> -acetyl-6- <i>O</i> - <i>tert</i> -butyldimethyl-silyl- β -cyclodextrin	Unspecified	0.25
iQ stainless steel capillary columns	Quadrex	WCOT	All phases	All	All
NTZB	VICI Gig Harbor	WCOT	Methyl polysiloxane	Various	0.1–0.53
Optima 624 LB	Macherey-Nagel	WCOT	6% cyanopropylphenyl-dimethyl-polysiloxane	1.8	0.32
Rtx-Dioxin2	Restek	WCOT	Proprietary	0.18, 0.25	0.18, 0.25
Rtx-XLB	Restek	WCOT	Proprietary	0.1–1.5	0.18–0.53
Select Al ₂ O ₃	Varian	PLOT	Al ₂ O ₃ MAPD	5	0.32, 0.53
SFC columns	Princeton Chromatography	Packed	Variety	N/A	2–4.6 (analytical); 10–50 semipreparative)

Length (m)	Comments
30, 50	Recommended for permanent gases and light hydrocarbon analyses (especially C ₁ -C ₆) in the petrochemical and chemical industries; deactivated with sodium sulphate or potassium chloride, the latter especially suited for common chlorofluorocarbon compounds; the alumina combined with the deactivation agents ensures that unsaturated hydrocarbons are eluted after saturated hydrocarbons and provides for specific elution patterns; improved water durability; columns are individually tested for efficiency, inertness and retention; chromatogram supplied; 180/200 °C upper temperature limits.
Various	A miniaturized nanofabricated packed capillary column module with integrated on-column thermoelectric temperature control; replaces the column and oven of conventional gas chromatographs; three models: general, air-cooled and subminiature for pocket GC systems; equivalent column length 2–6 m with 1 m increments; available with more than 400 stationary phases; operational from –5 to 200 °C; provides temperature ramps with heating rates as high as 300 °C/min; size of oven is 1/1000 that of a conventional oven and weighs 3–7 oz; applications include indoor air monitoring, personal medical monitoring, detection monitoring, personal medical monitoring, detection of explosive and illegal drugs and environmental monitoring.
20	Speciality column recommended and tested for high-speed separations of flavour–fragrance volatiles, especially lavender oil, whose retention indices can vary with temperature; 100 mm inner diameter reduces run times by one-third of previous 60 m × 0.25 mm column; other dimensions available; 250 °C upper temperature limit; phase ratio: 125.
15, 30, 60	Built-in deactivated guard column can be supplied with analytical stationary phases (VF-1ms, VF-5ms and VF-Xms) in FactorFour analytical column; no column coupling necessary; includes an integrated transfer line to MS systems, which results in lower bleed and stabilization.
15, 30, 60	Recommended for environmental and chemical applications in trace analysis; useful with GC–MS; low bleed, inert column for aggressive samples; phase ratio: 150–300; 350 °C temperature limit.
15, 30, 60	Recommended for routine analysis in environmental GC–MS applications; lower bleed than current columns; inert column shows no decomposition on analysis of <i>p,p'</i> -DDT; phase ratio: 150–300; 360 °C upper temperature limit.
5–6	Speciality column recommended for simulated distillation; special cross-linking to extend upper temperature to 450 °C; low bleed profile at high temperatures; phase ratio: 90–150.
Popular	A speciality column recommended for separation of <i>cis</i> – <i>trans</i> fatty acid methyl esters using GC–MS; arylene backbone provides increased thermal stability and coating stability compared with conventional siloxane phases; temperature limits: 250/260 °C; specially tested for <i>cis</i> – <i>trans</i> hydrocarbon resolution.
25, 50	A speciality column for the enantiomeric separation of flavour compounds including underivatized alcohols, esters, ketone, aldehydes and δ -lactones; no requirement for derivatization of alcohols saves time and money; temperature limits: 220/240 °C.
All	All of the company's standard phases, including thick films, are available in stainless steel capillary versions; columns use new Inert passivation technology; columns are designed to be rugged and unbreakable; applications include process GC, portable GC systems and standard GC systems.
5–60	NTZB = next-to-zero bleed; general-purpose column with upper temperature limit of 450 °C with low bleed; no change in film thickness at maximum operating temperature.
30, 50	A low-bleed version of the company's standard Optima 624 column; recommended for environmental analysis according to US EPA Method 624 for halogenated compounds and other volatiles; thick-film, low-bleed version provides higher detection sensitivity.
40, 60	Speciality column recommended for separating dioxin and furan congeners; resolves 2,3,7,8-dioxins from each other and other congeners and furans from chlorodiphenyl ethers; upper temperature limit: 320 °C.
15,12–60	Recommended for separating semivolatile environmental pollutants and high molecular weight analytes; temperature limits: 340/360 °C; less than 6 pA bleed at 320 °C; useful with GC–MS methods and for US EPA Method 525.2.
50	MAPD = methyl acetylene and propadiene, reactive and difficult to quantitate at low levels; recommended for all MAPD C ₁ –C ₅ hydrocarbons at trace levels; as much as a factor of 2 higher response for polar hydrocarbons such as propadiene and methyl acetylene, which results in greater accuracy and better QC data; upper temperature limit: 200 °C.
0.05–0.25	Analytical and semipreparative SFC columns packed with either 5 μ m or 10 μ m particles; many stationary phases including ones specifically developed to enhance SFC separation; columns are individually tested by SFC.

Table 2: Sample preparation products at Pittcon 2004.

Product Name	Supplier	Product Type	Mode(s)	Base Material	Functional Group
48-well SPE plate	United Chemical Technologies	SPE plate	Various	Silica	Various
Bioanalytical Toolbox	Argonaut Technologies	Biological fluid extraction kit	SPE, protein precipitation; support liquid extraction; filtration	Silica (for SPE)	C2, C8, C18, C8/sulphonic, C8/quaternary amine
Chromabond ABC 18	Macherey-Nagel	SPE column	Reversed phase/ion exchange	Unspecified	Unspecified
Chromabond Phase Separation columns	Macherey-Nagel	Phase separation column	Extraction	Special membranes	N/A
Dioxin Sample Prep system	Supelco	System	Adsorption	Silica	Silica
Discovery DSC-MCAX SPE	Supelco	SPE	Mixed	Silica	C8, benzenesulphonic acid
EluChrom extraction cartridges	Analytical Sales and Service	Extraction cartridge	Matrix solid-phase dispersion	Polymer	Unspecified
Empore deep well plates	3M Bioanalytical	SPE 96-well plate	Reversed phase	Silica	C2, C8, C18
Empore filter plate	3M Bioanalytical	96-well filter plate	Filtration	Polypropylene	N/A
IC Millex filter units	Millipore	Syringe filters	Filtration	Polypropylene, PTFE membrane	N/A
iLAP plates	Sigma-Aldrich	96-well plate	Cell lysis and affinity	Polypropylene construction	Cell lysis reagent-HIS chelate matrix
Isolute 103	Argonaut Technologies	SPE cartridge-96-well plate	Reversed phase, hydrophobic interaction	Hydroxylated PS-DVB	Hydroxylated PS-DVB
Isolute PE-AX/SCX-2	Argonaut Technologies	SPE cartridge-96-well plate	Ion exchange	Silica	Sulphonic acid and quaternary amine
LC Fiber	Glygen	SPE capillary	Various	Polypropylene or fluorinated ethylene propylene	Various

Dimensions	Comments
96-well plate size	Solvent-resistant plate holds 48 disposable cartridges that can be packed with a variety of functionalized silica gels; design accommodates a larger sample volume than the standard 96-well plate; collection directly into collection vials for further handling; can be used with liquid-handling systems such as the Bohdan miniblock and Tecan Automated Parallel SPE purification systems or with standard house vacuum; two versions available.
96-well plate size	Four-sample preparation techniques (including filtration) in one kit; non-polar and mixed-mode SPE provides broad selectivity; recommended for bioanalytical sample preparation method development to provide the most suitable procedure for optimum analyte recovery and extract cleanliness; can be used with sample-processing stations or automated liquid-handling systems.
500 mg/6 mL	Packed polypropylene column for the clean-up of acrylamide from ultraheated starch-containing food; eliminates compounds that can contaminate MS sources or decrease sensitivity; clean-up protocols available.
1–70 mL	Phase-separation columns constructed of polypropylene: PTS is for solvents heavier than water, PTL for solvents lighter than water.
350 mm × 17 mm	Clean-up system for dioxin analysis; glass column prepacked with seven layers of silica gel treated with reagents such as silver nitrate, sulphuric acid or potassium hydroxide combined with separate conditioned, dual-layer (Carboxen 1016, Carboxen 1000) carbon reversible tube; rapid procedure saves 1–2 days per sample; suitable for dioxin analysis under US EPA Method 1613; vacuum manifold, fittings and other components are available in kit form or separately; packings are available in bulk.
Unspecified	Mixed-mode cation exchanger and reversed-phase SPE packing available in cartridges and 96-well plates; uses both hydrophobic and electrostatic interactions to retain acidic, basic, neutral and zwitterionic compounds from aqueous solutions; selectivity can be tuned by adjusting mobile-phase conditions such as pH and organic solvent composition.
3, 12, 25 and 70 mL cartridges	Recommended for the extraction of drugs from solid biological samples such as tissue; device is a syringe barrel that functions as a dual-compartment SPE cartridge with two independent beds; the lower bed is sandwiched between frits; the 3 g top bed consists of loose sorbent that extracts analytes from the tissue sample; the 1.5 g bottom bed separates the components extracted in the top compartment.
96-well plate size	Polypropylene deep-well plates have a 2.5 mL volume, which is double the capacity of current 1.2 mL plates; compatible with standard vacuum manifolds and automated liquid-handling systems.
96-well plate size	Standard 96-well plate with 1.2 mL capacity; has built-in filters of different density starting with the coarsest on the top and the finest on the bottom, which reduces plugging and improves filtering efficiency; used for protein precipitation with acetonitrile (200:800 mL ratio); can be used with any vacuum manifold or automated liquid-handling system.
13 and 25 mm	PTFE membranes with 13 mm and 25 mm diameters; volume handled: 1–10 mL for the 13 mm membrane and 10–100 mL for the 25 mm version; low extractables provide cleaner chromatography; low binding; polyethylene housing; units individually packaged.
96-well plate size	Recommended for rapid colony screening and studies of protein–protein interaction; integrated lysis and affinity purification (iLAP) plate combines cell lysis and HIS-tagged protein-purification steps into one step; 1 mL and 2 mL well plates include lysis reagent and affinity matrix; lysis component includes lysozyme for cell lysis of both Gram-positive and Gram-negative bacteria and protease inhibitors to prevent proteolytic degradation of target proteins and Benzonase to clarify viscous DNA fragments; high capacity allows capture of at least 4 mg of HIS-tagged protein per well; <i>BIO</i> .
Cartridge: 200 and 500 mg/3 mL or 6 mL; 96-well: 200 mg/2 mL well	Recommended for isolation of organic reaction products from semipreparative LC organic solvent–water mobile-phase fractions; capturing analyte on the SPE phase eliminates evaporation of solvent and saves time; tabless cartridges and 96-well plates for automation; surface area: 1000 m ² /g; particle size: 90 µm.
Cartridge: 200 and 500 mg/3 mL or 6 mL; 96-well: 200 mg/2 mL well	Recommended for isolation of acidic and basic starting materials and impurities from organic reactions that have neutral molecules as target compounds; a single column removes both acid and basic impurities; tabless cartridges and 96-well plates for automation; particle size: 40–60 µm; 60 Å pore size; ion-exchange capacities: PA-AX = 0.55 mequiv/g, SCX-2 = 0.45-mequiv/g.
1–50 cm × 50–500 µm	Capillary tubes for sample preparation in which the chromatography material is attached directly to the inner surface of the capillary; the material can be any chromatography packing; tubes can be used for capillary LC and CEC or as HPLC sample loops for precolumn purification.

Table 2: Sample preparation products at Pittcon 2004 (continued).

Product Name	Supplier	Product Type	Mode(s)	Base Material	Functional Group
Microlute 96-well development plate	Porvair	SPE 96-well plate	Many	Silica, polymer	See comments
MP-TsOH	Argonaut Technologies	SPE cartridge	Ion exchange	Macroporous PS-DVB	Sulphonic acid
Multiple Affinity Removal system	Agilent Technologies	Immunodepletion column	Affinity	Proprietary modified polymer	Various affinity ligands
MultiScreen filter plates	Millipore	96-well filter plate	Filtration	Polypropylene, polycarbonate, PTFE	N/A
NuTip/TopTip/SyringeTip	Glygen	SPE tip	Reversed phase	Polypropylene tip and packing	C18
Oil and grease extraction disc	3M Bioanalytical	SPE disc	Reversed phase	Silica	C18
OMIX pipette tips	Varian	SPE tip	Reversed phase	Silica monolith	C18
PL EnviroPrep	Polymer Laboratories	Clean-up column	Size exclusion	PS-DVB	PS-DVB
Sirocco protein precipitation plate	Waters	96-well plate	Precipitation	Unspecified	N/A
SPE Mixed Mode Drug-Clean tubes	Alltech Associates	SPE cartridge and 96-well plate	Reversed phase-ion exchange	PS-DVB or silica	Unspecified reversed-phase and cation exchanger
Strata-X On-line extraction column	Phenomenex	On-line SPE cartridge	Proprietary	PS-DVB	Proprietary polar functionalized
Titan2 syringe filters	Sun SRI	Syringe filter	Filtration	Polypropylene plus membrane	N/A

for diagnostics purposes and other uses. In human plasma, there are six highly abundant proteins that make up 85% of the proteins present. Often in gel

electrophoresis and HPLC studies, these proteins dominate the minor proteins of interest and hinder their study. Agilent Technologies has introduced an affinity-

based product in a column format that will remove the high-abundance proteins and elute the remaining proteins for further study. An example of such an application is

Dimensions	Comments
96-well plate size	Recommended for high-throughput screening method development; available with Argonaut and Varian sorbents for reversed-phase, normal-phase, ion-exchange and other modes, 10–100 mg/well; custom-packed plates available; starter pack has several different sorbents, manifold and accessories.
See comments	Recommended for isolation (scavenging) of basic impurities or target compounds (catch and release) from organic reaction mixtures; retained basic compounds are eluted with ammonia–methanol; improved capacity resin compared with silica-based ion-exchange sorbents; sizes (tabbed and tabless): 100 mg/3 mL, 250 mg/6 mL, 500 mg/6 mL; sizes (tabbed): 1 g/15 mL, 2.5 g/25 mL; 24- and 48-well plates available.
50 and 100 mm × 4.6 mm	Immunodepletion affinity column for removing six high-abundance proteins — albumin, IgG, IgA, transferrin, haptoglobin and antitrypsin — from human biological fluids for proteomics investigations so that low-abundance protein biomarkers can be more easily identified; HPLC column format, custom column sizes available on request; <i>BIO</i> .
96-well plate size	Variety of speciality plates: MIC plates are optimized for migration invasion and chemotaxis assays, with a choice of 3, 5, or 8 µm polycarbonate membranes; Solubility filter plates for quantitative solubility assays incorporate low-binding membrane and plate materials to yield high drug recovery; Solvintert filter plates are specially designed for broad solvent compatibility with PTFE membranes; the plates are automation compatible and can be used with aqueous and non-aqueous solvent systems.
Pipette tip	Micropipette tip for preparation and separation of biological samples in microlitre and nanolitre volumes; can be used for desalting samples; chromatography material is attached to the inner surface of the tip; two sizes: 1–10 µL and 10–200 µL; can be used with samples as small as 100 nL; SyringeTip version attaches to a microlitre syringe; TopTip has chromatography material loosely packed into the tip with a very fine slit at bottom; <i>BIO</i> .
47- and 90-mm discs	Recommended for an alternate performance-based approach to US EPA Method 1664 : <i>n</i> -Hexane Extractable Materials and Silica-Gel-Treated <i>n</i> -Hexane Extractable Material by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons); method applicable to aqueous matrices that requires the use of <i>n</i> -hexane as the extraction solvent and gravimetry as the determinative technique.
Pipette tip	Recommended for microextractions for peptide–protein purification in proteomics applications; small bed of functionalized monolithic sorbent inserted inside a pipette tip; improved flow characteristics; elution volume range: 2–10 µL; MB version is mini-bed with elution volumes of 0.5–2.0 µL; <i>BIO</i> .
150 and 300 mm × 25 mm	Recommended for clean-up of environmental samples; 100 Å pores; 10 µm particle size; non-aqueous size-exclusion mode; column is constructed from stainless steel.
96-well plate size	Recommended for in-well protein precipitation from biological samples; plate includes a self-contained filter system, a sealing cap mat and a built-in vacuum-activated valve that prevents clogging, cross-contamination and leaking during use.
Variety of weights and volumes	Recommended for drug of abuse, with protocols provided; mixed-mode (copolymeric packing) extraction from biological fluids with a choice of highly crosslinked PS–DVB polymer base material or silica base material with reversed-phase and cation-exchange groups polymerized on the base material; the polymer base material's high capacity enables the use of a small amount of packing per well for 96-well plate; particle size: 40–60 µm; pore size (for silica): 60 Å.
20 mm × 2 mm	Stainless steel column recommended for on-line SPE extraction, concentration and separation of drugs in biological matrices; cartridge can be used as a stand-alone device; reusable for more than 200 injections; direct injection of biological fluids; detection limits of 100–200 pg; separation times in the range of 1.2–3 min/sample.
4, 17, 30 mm	New sizes; autoclavable; colour-coded for easy membrane identification; porosities: 0.20 and 0.34 µm; sample volume: <1 mL for the 4 mm filter (retention volume is <7.5 µL), <10 mL for the 17 mm filter (retention volume is <25 µL), 2–100 mL for the 30 mm filter (retention volume is <115 µL); operating pressures: 75–100 psi.

depicted in Figure 2. The sample is passed through the column, and low-abundance proteins are eluted within 4 min, while the covalently bonded antibodies on the column

hold the high-abundance proteins. Then, with a stronger buffer, the high-abundance proteins are eluted in the 10–11 min time period, and the column is regenerated for

the next sample. The low-abundance proteins can be further concentrated using a spin tube with a reversed-phase packing, an SPE cartridge or a pipette tip.

Table 3: Chromatography and sample preparation accessories and kits at Pittcon 2004.

Product Name	Supplier	Application Area	Product Type
96-well vacuum manifold assembly	3M Bioanalytical	SPE, filtration	Vacuum manifold
Active splitter for LC-MS	Rheodyne LLC	LC-MS	Flow splitter
CE-100 SA Zero Flow capillaries	MicroSolv Technology	CE, CEC	Polymer-coated CE capillary
Celerity CE	MicroSolv Technology	CE, CEC, OT-CEC	Bonded-phase capillaries
Chromatography Column Rodaviss joints	Kimble Kontes	LC	Glass columns
Chromolith guard cartridge	Merck/EM Science	LC	Guard column
Confluent fluidics products	Scivex	LC	Pumps and valves
CRD-1 carbonate removal device	Dionex	LC	Membrane device
EOTrol Dynamic coatings	Target Discovery	CE, CEC, microfluidics	Dynamic coating
FlipTop inlet sealing system	Agilent Technologies	GC	Inlet liner changing system
Glycoprotein analysis solutions	Sigma Aldrich	LC, CE	Purification and modification
Glyphosate analysis kit	Pickering Laboratories	Sample preparation, LC	Herbicide analysis kit
Guardian	Phenomenex	GC	Integrated guard column
HD Crimper	Chromatography Research Supplies	Sample preparation-handling	Crimper-decapper tool
Hot Sleeve	Analytical Sales and Service	LC	Column heater
MassPREP standards	Waters	LC, LC-MS	Peptide standards
Mycotoxin analysis kit	Pickering Laboratories	Sample preparation, LC	Multiresidue screen kit
Nano-Injector	Dychrom	LC	Nano-injector
Nano-Scale valves	Rheodyne LLC	LC	Injection and switching valves

Suggested Applications	Comments
SPE and filtration	Recommended for use with 96-well plates from 3M or other manufacturers; transparent top; replaceable gaskets; chemically resistant materials.
Split flow to MS source	Active splitter performing mass rate attenuation; user programmable splits of 1:100 to 1:100 000; periodically gates small aliquots to MS system's analytical stream.
To eliminate EOF in CE	Polymeric coating is chosen to eliminate electroosmotic flow (EOF) so that analytes separate according to mobility differences; pH resistant to pH 8.5; dimensions: 100 cm × 50 and 75 µm; capillaries are polyimide-coated on the outside; compatible with all CE instruments; <i>BIO</i> .
Protein separations by CE	Celerity CE bonded capillaries with new C18-cholesterol, C8 and biphenyl-coated phases; polyimide-clad; 20 µm inner diameter for high resolution; <i>BIO</i> .
Flash chromatography	Gravity glass columns feature Rodaviss joints that provide air-tight seals and allow disassembly of frozen joints because of residue formation; columns have fritted discs, PTFE stopcock plugs; available in a variety of sizes; custom columns and plastic safety coating available.
Protects analytical column	Chromolith silica monolith guard-cartridge kit with three guard cartridges, holder and wrench; kit dimensions: 5 and 10 mm × 4.6 mm ; RP-18e stationary phase.
Nano- and capillary LC	Nano fluidic module delivers isocratic flows at extremely low flow-rates for MS sample injection, lab-on-a-chip infusion and trap-column regeneration; nanopeak valves have fittings for 1/32 in. o.d. tubing, pressure-rated to 5500 psi in six- and seven-port selection valve formats; rotary selenoid valves and precision dispensing pumps also available.
Carbonate removal from mobile phase	Designed for use with the Dionex Hydroxide Eluent Generator and for anion-exchange separations in which carbonate is a major sample-matrix component; removes carbonate by adsorbing carbon dioxide through a permeable membrane into a basic scrubber solution; installed upstream from the conductivity cell; useful for inorganic anion determinations at trace levels; applications include drinking water and ground water.
To control EOF	A line of dynamic coatings that control EOF in capillaries and microfluidic electrophoretic systems; the coatings are nearly pH- and buffer-independent; the coatings are added to buffer in bare capillary systems between pH 2 and pH 12, and users select the type of EOF control; the coatings are stable to chaotropic agents and surfactants; <i>BIO</i> .
Quick change of inlet liner	Allows quick and easy access to inlet liners for more efficient liner removal and replacement; has lever arm assembly that locks to the injector port; allows change of an inlet liner in 30 s without burning fingers, scraping knuckles or searching for tools.
Glycoproteins	Kits available for in-gel or in-solution enzymatic protein deglycosylation and trypsin digestion, glycoprotein gel-membrane staining and in-gel detection, native protein deglycosylation and enzymatic protein deglycosylation; all reagents supplied; <i>BIO</i> .
Glyphosate and AMPA in crops	Kit contains cation-exchange columns and reagents for clean-up of glyphosate and AMPA in crops according to AOAC Method 2000.52; measurement by postcolumn reaction system with fluorescence detection.
Protect column against contaminants	Guard column system is deactivated for inertness; temperature limits: 360/370 °C; designed to provide better sealing than traditional guard columns.
Crimping GC and LC vials	Electronic crimper with interchangeable jaw sets for 8, 11, 13 and 20 mm crimp caps including steel and magnetic caps; recommended for high-volume or pilot-scale operations; can be mounted via a support bracket or used as a portable tool when working with vials in racks; has a controller box and an optional foot pedal.
Heating LC columns	Tube-style design that encapsulates columns as long as 30 cm; temperature controller has fast 10 Hz sampling that improves control responsiveness; also has variable-burst firing that prolongs heater life and provides better temperature control; external events inputs can disable temperature control for shut-off.
Method development and validation	Peptide standard mixture and protein digest standards help scientists develop and validate bioanalytical methods for HPLC and LC-MS. MALDI matrices are also available for LC-MALDI-MS; <i>BIO</i> .
Aspergillus and Fusarium mycotoxins	Kit consists of a sample SPE affinity column, a reversed-phase column, postcolumn derivatization reagents and methodology for mycotoxin determination in peanuts and wheat according to AOAC Method 991.31.
Capillary and nanocolumn injection	Developed by Chemco Scientific; 20 nL internal channel; recommended for use with columns with inner diameters as small as 100 µm; direct-connect system with male threads provides low dead volume.
Automated HPLC injection and column switching	Two-position, four-port motorized injector with internal 10 nL sample loop with 0.10 mm internal passages; two switching valves, with six and 10 ports, 0.10 mm flow passages, and port-to-port volume less than 25 nL; can be used for stream selection, on-line sampling and column selection.

Table 3: Chromatography and sample preparation accessories and kits at Pittcon 2004 (continued).

Product Name	Supplier	Application Area	Product Type
Precut fused-silica tubing	New Objective	LC, CE	Fused-silica tubing
ProteCol flowmeter	SGE	LC, LC-MS	Flowmeter
Protein digest calibration standards	MicroSolv	LC, LC-MS, CE	Peptide standards
Radial diaphragm valve	Swagelok	LC	Radial diaphragm valve
Sealing tape pads	3M Bioanalytical	SPE, filtration	96-well plate
SPE-Dex extraction system	Horizon Technology	SPE	Automated SPE system
Symbiosis Pharma	Spark Holland	SPE-LC	On-line SPE system
UltraClean Plasmid kits	MoBio Laboratories	Sample preparation	Spin filter; 96-well plate
VersaFlash	Supelco	LC	Flash chromatography system

Hardware and Accessories

Table 3 lists 28 new hardware products and accessories for HPLC, GC, CE and sample preparation. This year, I have extended the columns and sample preparation accessories to include various kits that are designed to solve specific application problems. All of the products are practical devices designed

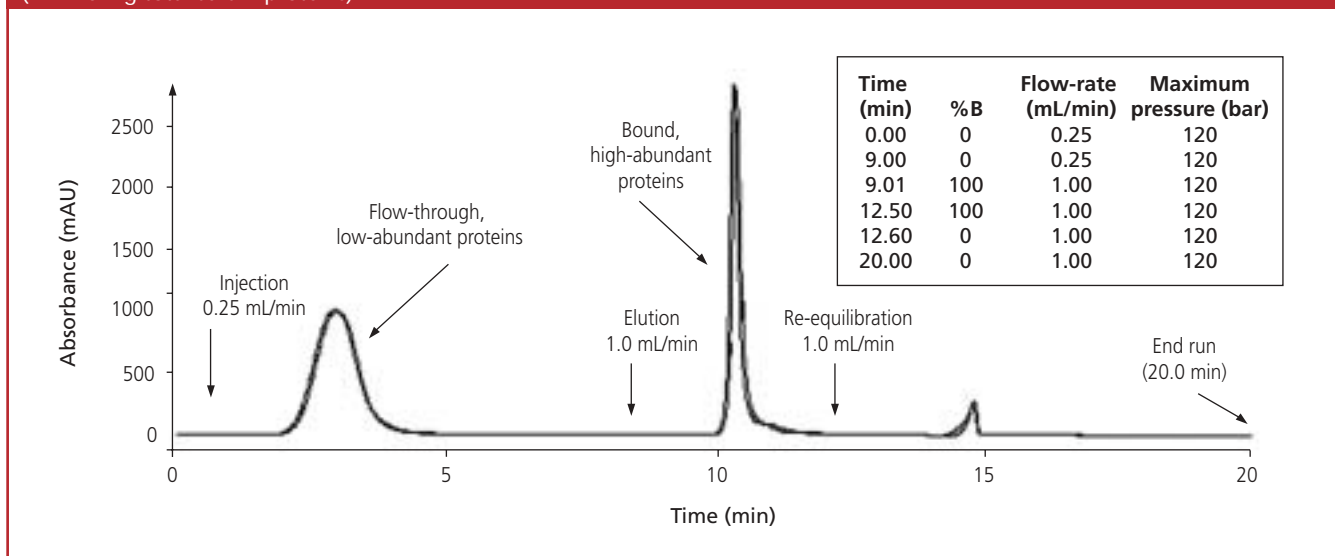
to make chromatographers' lives easier. I will cover each area individually.

HPLC and ion chromatography products:

With the advent of capillary- and nano-LC and LC-MS needs, component manufacturers such as New Objective, Rheodyne and Scivex (formerly Upchurch Scientific) have developed small injectors,

valves, connectors, precut tubing, columns, flow splitters and interfaces to fill these niches. Guard columns are always popular, and many new guard columns to protect valuable analytical and preparative columns have been introduced both as stand-alone (discussed here) and for the various modes (shown in other sections). Dionex has

Figure 2: Comparative chromatograms from runs 20 and 200 of immunoaffinity separations of low- and high-abundance proteins in serum (Agilent Technologies). Column: 50 mm × 4.6 mm Multiple Affinity Removal; injection volume: 15–20 µL of serum (1.2–1.6 mg total serum proteins).



Suggested Applications	Comments
Capillary connections	Precut tubing with machine-polished ends for leak-free connections; for plumbing valves and HPLC and CE connections; 6, 10, 15, 25 and 50 cm × 20, 50, 75 and 100 µm; outer diameter: 360 µm.
Flow measurement in capillary LC	Operates with any LC system; designed to help achieve reliable, reproducible capillary flow, especially when a conventional pump and flow splitter is used to obtain low flow-rates; simple and direct measurement; two configurations, one optimized for flows of 0.5–5 µL/min and the other for flows of 5–50 µL/min.
Instrument calibration and method development	High-purity dithiothreitol-reduced IAA-alkylated proteins digested with TPCK-treated trypsin; available individually or in kits of low and high molecular weight proteins; <i>BIO</i>
High pH, high temperature	Radial diaphragm valve now available with polyethersulphone actuator that offers resistance to caustic wash-down and can withstand higher temperatures than polysulphone; available in many sizes and can be manufactured with various configurations.
Sealing of 96-well plates	Designed to seal unused portions of 96-well plates only, not for sealing collection plates.
EPA methods, pharmaceuticals	A fully automated sample preparation system for rapid extraction of organic chemicals from aqueous samples; recommended for EPA Methods 500, 600 and 8000 plus polychlorinated biphenyls, diesel-range organics, total petroleum hydrocarbons, oil and grease, pesticides, herbicides and explosives.
Drugs in biological fluids	Dedicated on-line SPE system for automated, high-throughput LC–MS with approximately 50 standard sorbents available; recommended for use in bioanalysis, toxicology and early absorption, distribution, metabolism and excretion studies; improved performance over off-line SPE approaches.
Isolation of plasmid DNA	Mini Plasmid Prep kit purifies high-quality, supercoiled plasmid DNA in 6 min using a spin filter to bind plasmid DNA; yields as much as 20 µg of plasmid from 1–5 mL of culture; 96-well kit processes 96 samples in 45 min; large-scale kits are also available; contains all necessary reagents and tubes; <i>BIO</i> .
Organic synthesis product purification	Uses disposable flash chromatography silica cartridges; cartridges are stackable and reversible; scalable to more than 70 g.

extended its line of on-line ion-chromatography devices with the introduction of a carbonate-removal module. This carbonate-removal device, used with the company's hydroxide eluent generator, is a new concept in handling samples with high concentrations of carbonate for use in anion-exchange applications. Figure 3 shows a schematic of the carbonate-removal device. The key to its functionality is the use of an extremely thin film of carbon dioxide-permeable polymer on the exterior surface of a narrow-bore asymmetric capillary membrane. On one side of the membrane is the carbon dioxide-containing fluid, and on the other side of the membrane a basic solution flows in a countercurrent manner. More than 95% of the carbon dioxide can be removed. Figure 4(a) shows a chromatogram from an analysis of a control sample of drinking water (1000 µL injection) containing a large concentration of carbonate without the carbonate-removal device installed. Note the presence of the carbonate peak at the 8 min timepoint. With the carbonate-removal device installed, most of the carbonate is removed [Figure 4(b)].

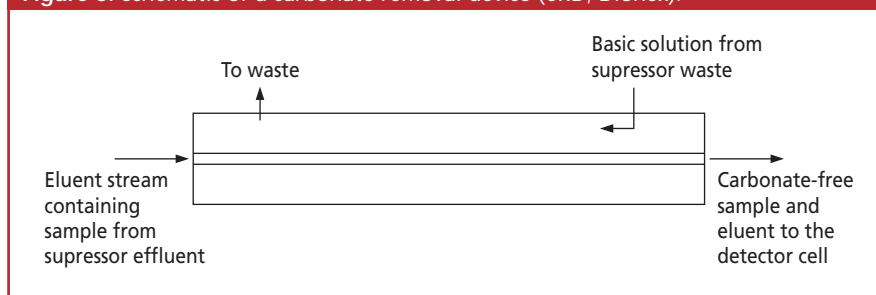
GC and SFC products: Everyone hates changing liners in their gas chromatograph because invariably, they either burn their

Guard columns are always popular, and many new guard columns to protect valuable analytical and preparative columns have been introduced both as stand-alone (discussed here) and for the various modes (shown in other sections).

fingers attempting to work with a hot injector port or scrape their knuckles while undoing septum nuts. A new flip-top sealing system has made the whole process easier. Guard columns for GC are just as valuable as they are for LC, and Phenomenex has developed an integrated guard column to protect the analytical column against high boilers and other low-volatility substances.

Sample preparation products and analysis kits: A number of kits have been assembled to prepare and analyse specific analytes such as glycoproteins, plasmids, glyphosate and mycotoxins. Normally, these kits consist of everything needed for the sample preparation and usually an LC column for the analysis. Some of the kit suppliers even provide prepared reagents. Calibration standards for peptide analysis

Figure 3: Schematic of a carbonate removal device (CRD, Dionex).



were another popular accessory this Pittcon. Automated and semiautomated SPE systems were introduced to meet the demands of high throughput.

Capillary electrophoresis (CE) and capillary electrochromatography (CEC)

products: CE is alive and well and new products continue to be developed to solve old problems. The new products introduced by MicroSolv Technology and Target Discovery mainly control the electroosmotic flow inside of the capillary to make separations more controllable and reproducible.

References

1. R.E. Majors, *LC•GC Eur.*, **17**(4), 200–215 (2004).

Acknowledgement

I would like to thank the manufacturers and distributors who kindly furnished the requested information in advance of Pittcon 2004, thus allowing a timely report of new product introductions. For those manufacturers who would like to be considered for inclusion into Pittcon 2005 coverage, please send the name of the primary company contact, mailing address, fax number and e-mail address to "Column Watch" Pittcon 2005, *LC•GC Europe*, Advanstar House, Park West, Sealand Road, Chester CH1 4RN, UK or e-mail dhills@advanstar.com

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