DAISOGEL™

Packing Materials for Process Purification

Waters
THE SCIENCE OF WHAT'S POSSIBLE™
DAISOGEL
Packing Material for Process Purification
- offered by Waters
Waters Corporation and DAISO CO., LTD., Osaka, Japan, have jointly entered into a collaboration to globally market and supply DAISOGE™ bulk packing materials for process chromatography.

By combining the purification expertise of both organizations, pharmaceutical and biopharmaceutical companies’ scaling processes for lab-scale to pilot plant or process-scale manufacturing, can now benefit from an unparalleled level of technical and product support.

**DAISOGE Packing Materials**

- Silica-based packing materials for pharmaceutical and biopharmaceutical chromatographic purifications
- High mass loading and excellent resolution
- Excellent durability with extended acidic and alkaline resistance
- Available in multiple kilograms

**DAISOGE Analytical and Preparative HPLC Columns Packed by Waters**

- Analytical columns for screening and method development of isolation and purification methods
- Preparative columns packed with Waters patented Prep OBD™ Technology for scaling up
COLUMN PERFORMANCE – IDENTIFYING THE PROBLEM

Laboratory-scale HPLC purification presents many challenges to the chromatographer. Inconsistency in column-to-column performance and lifetimes often result in lost samples, repeat purification runs and poor scalability from small to larger volume columns. Common HPLC prep column failures are due to mechanical instabilities, especially voids in the packed bed. The patented OBD Technology\(^1\) combines optimized packing processes with a unique set of column designs ensuring increased lifetime and better reproducibility.

When a column is used in a preparative HPLC system, it must remain stable during operation. The bed must be packed sufficiently dense to withstand the compressive fluid forces encountered during use. In the case of analytical column dimensions, the necessary packed bed density can be generally achieved using traditional slurry packing methods. As the diameter of the column increases, it becomes increasingly difficult to reach the bed density required for stable, long-term performance, especially when using small particles. The optimum bed density is dependent upon the specific properties of the chromatographic particles and column dimensions.

\(^{1}\)US Patent # 7,399,410; UK Patent # GB2408469

OBD COLUMN DESIGN – IMPROVED MECHANICAL STABILITY

Waters combines high-pressure slurry packing with a carefully calculated axial compression element localized at the less-dense inlet end of the bed. With careful tuning of the packing process for each particle type and column geometry, the Prep OBD design and process results in predictable, uniform density profiles throughout the column. Established procedures ensure that the column bed is not over-compressed or disrupted during the final column packing process. Excess axial compression applied at the inlet can break particles, build bridges, and lower local bed permeability.

Exploded Prep OBD Column Hardware

The patented OBD column hardware design incorporates chemically inert seals made to prevent leaks and a pair of specially designed filter housings with frits and distributors built to withstand high pressures.
DAISO CO., LTD.

DAISO CO., LTD., headquartered in Osaka, Japan, is a technology-oriented chemical company supporting industries including medicine, agriculture, IT, machinery, textiles, and construction through innovative products.

PRODUCT OFFERING INCLUDES:

- DAISOGEL packing materials
- Pharmaceutical intermediates
- Fine chemicals
- Proprietary functional chemicals
- Soda products and Chlor-Alkali
- Synthetic and fabricated resin products

www.daiso-co.com

DAISOGEL PACKING MATERIAL

DAISO CO., LTD. is one of the leading silica gel manufacturers for liquid chromatography, using inorganic synthesis technology. Since 1992, DAISO has added large-scale manufacturing capability for DAISOGEL media to fully support the process purification laboratories worldwide. DAISOGEL phases are used for various applications including medicines and pharmaceuticals, agricultural chemicals, food ingredients, biomolecules, liquid crystals and functional dyes.

DAISOGEL packing materials have been designed to meet the needs of process purification. These spherical silica-based packing materials allow for high mass loading and maximum resolution which can result in reducing the number of repeated injections. Through high density bonding and endcapping, DAISOGEL packing materials have shown extended life and durability. These benefits have helped customers in reducing their overall purification costs.

QUALITY

DAISO quality control system is based on ISO 9001 for the silica gel manufacturing plant and has a DMF (Drug Master File) on file with the FDA (Food and Drug Administration) for the DAISOGEL packing materials.

Starting with the raw materials, the quality of DAISOGEL packing material is strictly controlled at each manufacturing step. Each DAISOGEL batch is analyzed for pore size, surface area, pore volume and particle size, along with measurements of the silica gel pH, volatile matter, and metal impurities. DAISOGEL packing materials are designed to perform with consistent selectivity from lot-to-lot and between particle sizes.
DAISOGEL Packing Materials

DAISOGEL SP-120-ODS-RPS
- Silica-based 120Å C18 phase
- High coverage and exhaustive endcapping

ODS-RPS packings featuring maximum surface coverage are the ideal choice for a wide variety of organic compounds. Carefully controlled full endcapping leads to optimal performance with acidic, basic and chelating compounds.

Analysis of Vitamin B

- Column: DAISOGEL SP-120-5-ODS-RPS 150 x 6 mm
- Eluent: 10 mM sodium phosphate (pH 5.0)/CH3CN (85/15)
- Flow Rate: 1.0 mL/min
- Detection: UV @ 254 nm
- Temperature: 40 °C

Compounds:
1. Thiamin hydrochloride (Vitamin B1)
2. Riboflavin (Vitamin B2)

DAISOGEL SP-120-ODS-BP
- Silica-based 120Å C18 phase
- Suitable for hydrophilic compounds separation
- Longer lifetime in aqueous eluents
- Different selectivity from ODS-RPS

ODS-BP phases are designed to show extended selectivity for hydrophilic and polar compounds which are either poorly or not retained on other phases. Typical applications are separations of biomolecules and metabolites such as oligosaccharides, amino acids, small peptides, nucleotides and organic acids. They are endcapped and show similar selectivity as conventional C18 phases when being used for separations of hydrophobic compounds with typical reversed-phase eluents. They also show stable base lines and high sensitivity even under neutral pH conditions and without buffer or counter-ion additives.

Analysis of Pyridine/Phenol

No change in selectivity or retention after 100 hours of washing

Analysis of Organic Acids

- Column: DAISOGEL SP-120-5-ODS-RPS 150 x 6 mm
- Eluent: 30 mM sodium phosphate (pH 2.5 with H3PO4)
- Flow Rate: 1.0 mL/min
- Detection: UV @ 210 nm
- Temperature: 40 °C

Compounds:
1. Malonic acid
2. Nortriptyline
3. Acetic acid
4. Lactic acid
5. Citric acid
6. Succinic acid

Analysis of Antioxidants

Showing good retention of ascorbic acid

- Column: DAISOGEL SP-120-5-ODS-BP 150 x 6 mm
- Eluent: 0.1 M phosphate buffer (pH 3.1)
- Flow Rate: 1.0 mL/min
- Detection: ECD
- Temperature: Ambient

Compounds:
1. Ascorbic acid
2. GSH
DAISOGEL SP-200-C8-P
- Silica-based 200Å C8 phase
- Recommended for highly hydrophobic samples

C8-P packing materials are bonded with octyl groups and are fully endcapped. They are recommended for compounds which are too strongly retained on C18 phases. 200Å material is widely used for larger molecules with higher hydrophobicity.

Selectivity Comparison of Three Daisogel Phases

DAISOGEL SP-120-P
- Ultra-high purity 120Å silica

P phases are based on spherical and totally porous silica gel with metal impurities specified to be less than 10 ppm each for Al, Fe, Ti and Zr. Due to its narrow particle size distribution, P series packing material for preparative chromatography enables higher column efficiencies, lower operating pressure and longer column lifetimes.

Using DAI SOGEL, scale-up is guaranteed by using the same production procedure for all grades.

P series packing materials for preparative use are available in 10 µm, 15 µm and 20 µm. High purity 40-60 µm spherical silica is a cost effective alternative for medium and low pressure chromatography.

Analysis of Fat-Soluble Vitamins under Normal Phase Conditions

Preparative grades packing material in the past have had a wide particle distribution. The advantage of P series packing material for preparative chromatography is that it enables higher column efficiencies and lower operating pressure due to its narrow particle size distribution.
DAISOGEL SP-300-BIO

- Suitable for bulky biomolecules, proteins
- 300Å pore size, narrow particle size distribution, ultra high purity totally spherical silica gel
- High density bonding for extreme performance
- Available types: SP-300-ODS-BIO, SP-300-C4-BIO

The improved high density bonding and full endcapping make it most suitable for general use and to separate or purify high molecular weight compounds, especially proteins. Because of significant improvement in durability, these novel phases exhibit excellent acidic and alkaline resistance. These can be used for an extended period of time under acidic mobile-phase condition and rinsed for regeneration with NaOH containing solvents. The C4 phase is recommended for compounds too strongly retained on ODS C18 phases.

Excellent Durability in Acidic Environment

**Accelerated Acidic Duration Test Condition**
- Mobile Phase: CH3CN/1.0% TFA aqueous (10/90), pH 1.0
- Temperature: 70 °C
- Purge Time: 3 hr

**Chromatographic Test Condition**
- Mobile Phase: CH3OH/H2O (35/65)
- Temperature: 40 °C
- Detection: UV @ 254 nm

**Compounds:**
1. Uracil
2. Methyl Benzoate
3. Toluene
4. Naphthalene

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**DAISOGEL SP-300-C4-BIO**

250 CV Purged

**Competitor A (300Å, C4)**

250 CV Purged

**Competitor B (300Å, C4)**

250 CV Purged
Excellent Durability in Alkaline Environment

**Accelerated Alkaline Duration Test Condition**
Mobile Phase: CH$_3$CN/0.01 NaOH aqueous (10/90), pH 12
Temperature: 25 °C
Purge Time: 3 hr

**Chromatographic Test Condition**
Mobile Phase: CH$_3$OH/H$_2$O (35/65)
Temperature: 40 °C
Detection: UV @ 254 nm

**Compounds:**
1. Uracil
2. Methyl Benzoate
3. Toluene
4. Naphthalene

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**DAISOGEL SP-300-10-C4-BIO**

- Initial
- After 76 hr (25 cycles)
- After 19 hr (6 cycles)

**Competitor C (300Å, 15/20 µm, C$_4$)**

- Initial
- After 18 hr (6 cycles)
Ordering Information

DAISOGEL PRODUCT CHARACTERISTICS

- Exceptional batch-to-batch reproducibility
- Enhanced mechanical stability
- Suitable for Dynamic Axial Compression columns
- Narrow particle size distribution

GUIDE TO INTERPRETING DAISOGEL PRODUCT NAMES

- Silica particle shape: SP (spherical)
- Pore size (Å): 120, 200, 300
- Particle size (µm): 5, 10, 15, 20, 40/60
- Bonded phase: see table
- Base silica purity: P indicates ultra-high purity; Bio indicates ultra-high purity

DAISOGEL PHYSICAL PROPERTIES

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<th>Pore Size (Å)</th>
<th>Particle Size (µm)</th>
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ODS-RPS: High acidic resistance; suitable for organic compound separation
ODS-BP: low carbon content; suitable for hydrophilic and polar compound separation in up to 100% aqueous eluents
C8-P: for compounds too strongly retained on ODS phases
Silica: if no bonded phase is indicated, the product is bare silica
ODS-BIO: designed for biopharmaceutical applications; novel bonding technique ensures strong resistance against alkaline and acidic conditions
C4-BIO: new variation of the C4 phase; novel bonding technique ensures strong resistance against alkaline and acidic conditions
# DAISOGEL PACKING MATERIALS

## DAISOGEL COLUMNS PACKED BY WATERS

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For additional DAISOGEL products, please contact your local Waters sales representative.
Sales Offices

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All other countries:
Waters Corporation U.S.A.
1 508 478 2000
1 800 252 4752

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