

**Packed Columns for High Performance
Ion Chromatography
TSK-GEL IC Type**

INSTRUCTION MANUAL





TOSOH CORPORATION

Safety Precautions

To help protect your property from potential damage and ensure personal safety, please read this manual thoroughly before using the product.

[Notational Conventions]

Notation	Explanation
 WARNING	Alerts the user to the potential for serious injury or death.
 CAUTION	Alerts the user to the potential for damage to hardware or bodily harm.

WARNING

■ **Keep away from fire.**

Take proper precautions when using flammable solvents. There is the potential for fire, explosion, or poisoning.

CAUTION

■ **Use only in well ventilated areas.**

In case of insufficient ventilation, flammable and toxic solvents can cause fire, explosion, or poisoning.

■ **Do not spill solvents.**

Spillage and leakage can cause fire, electric shock, poisoning, injury, and corrosion. When cleaning up a spill, wear appropriate protective gear.

■ **Wear eye protection and protective gloves.**

Organic solvents and acids should not come in direct contact with the skin.

■ **Handle package with care.**

Inappropriate handling may cause rupturing and splattering.

■ **Only use this product as intended.**

This product is for separation and purification, do not use for any other purpose.

■ **Confirm compounds are safe.**

Check that obtained compounds and solutions after separation and purification are safe.

■ **Proper disposal.**

Dispose of in accordance with local laws and regulations.

NOTE

Keep this manual for future reference.

Contents

1. Introduction	1
2. Unpacking	1
3. General Information	2
3-1 Column Grades and their Specifications	2
3-2 Typical Applications	3
4. Configuration and Parts of Columns	4
5. Maintenance	5
5-1 Conditions of Measurement	5
5-2 Preparation of Eluent	6
5-3 Installation	6
5-4 Exchange of Solvents (Counter Ions)	8
5-5 Preparation of Sample Solution	8
6. Storage of Column	9
6-1 Protection from Drying and Freezing	9
6-2 Storage Conditions	9
7. Regeneration of Column	10
7-1 Clogging of Filter	10
7-2 Removal of Polyvalent Electrolytes	10
7-3 Removal of Hydrophobic Compounds	10
8. Calculation of the Number of Theoretical Plates and Asymmetry Factor	11
8-1 Method of Calculating the Number of Theoretical Plates	11
8-2 Method of Calculating the Asymmetry Factor	11
8-3 Dead Volume	11
9. Quality Specification and Warranty	12
9-1 Inspection Data	12
9-2 Quality Specifications	13
9-3 Warranty	13

1. Introduction

TSK-GEL IC is a packed column for high performance ion chromatography using no suppressing system developed by TOSOH CORP.

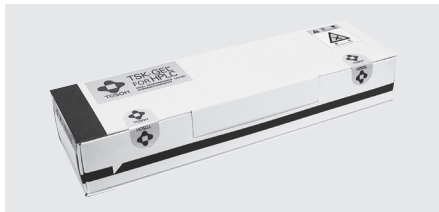
This column is featured by · Short column and high performance

· Applicable to a wide range of analytical conditions

This Instruction Manual contains crucial informations on how to care for and use these columns in the proper manner, so as to make most effective use of their high performance capabilities.

2. Unpacking

Check that nothing is the matter with the appearance of package or the column.



Appearance of the Package

Then check the following documents are attached to the column:

- 1) 1 copy Instruction Manual
- 2) 1 copy Inspection Data

3 . General Information

3-1 Column Grades and their Specifications

Column Grades and their Specifications are shown in Table 1 .

Table 1 Column Grades and their Specifications

Catalog Number	Column Grades	Column Sizes (mmID.xcmL)	Column Materials	Packing Materials	Particle Sizes (μ m)	Functional Groups	Ion-Exchange Capacities	Counter Ions	Packed Solvents for Shipping
06837	TSKgel IC-Anton-PW	4.6 x 5.0	Plastic	Polymethacrylate Gel	10	Quaternary Ammonium	30 \pm 3 μ eq/mL	Borate & Gluconate	Same Solvent *3 for Inspection
14463	TSKgel IC-Anton-PW _{XL}	4.6 x 3.5	SUS	Polymethacrylate Gel	6	Quaternary Ammonium	30 \pm 3 μ eq/mL *1	Borate & Gluconate	Same Solvent *3 for Inspection
06839	TSKgel IC-Anton-SW	4.6 x 5.0	Plastic	Silica Gel	5	Quaternary Ammonium	0.4 \pm 0.1 meq/g dry gel *2	Bitartrate	Methanol *4
07171	TSKgel IC-Cation	4.6 x 5.0	Plastic	Polystyrene Gel	10	Sulfonate	12 \pm 2 μ eq/mL *1	H ⁺	2mmol/L HNO ₃
08055	TSKgel IC-Cation-SW	4.6 x 5.0	Plastic	Silica Gel	5	Sulfonate	0.45 \pm 0.15 meq/g dry gel *1	H ⁺	Methanol *4
07172	TSKguardcolumn IC-A	4.6 x 5.0	Plastic	Polymethacrylate Gel	12	—	—	—	Same Solvent *3 for Inspection
07173	TSKguardcolumn IC-C	4.6 x 5.0	Plastic	Polystyrene Gel	10	Sulfonate	2.0 \pm 0.2*1 μ eq/mL	H ⁺	2mmol/L HNO ₃

*1 : Determined by titration in 0.5mol/L NaCl aq.

*2 : Estimated by elemental analysis of nitrogen.

*3 : 1.3mmol/L Gluconic acid + 1.3mmol/L Boric acid pH 8.5.

*4 : Exchange methanol into distilled water before column is connected with instrument

3-2 Typical Application

Typical applications are shown in Table 2.

Table 2 Typical Applications

Column Grades	Typical Applications
TSKgel IC-Anion-PW	Analysis of Anions such as F ⁻ , Cl ⁻ , NO ₃ ⁻ , Br ⁻ , PO ₄ ³⁻ , SO ₄ ²⁻ etc.
TSKgel IC-Anion-PW _{XL}	Analysis of Anions such as F ⁻ , Cl ⁻ , NO ₃ ⁻ , Br ⁻ , PO ₄ ³⁻ , SO ₄ ²⁻ etc.
TSKgel IC-Anion-SW	Analysis of relatively strong Hydrophobic Anions such as SCN ⁻ , S ₂ O ₃ ²⁻ , BrO ₃ ⁻ etc.
TSKgel IC-Cation	Analysis of Cations (Measure with metal free system)
TSKgel IC-Cation-SW	<ol style="list-style-type: none"> 1) Analysis of Heavy Metal Ions which contain transition metal ions such as Fe, Cu, Ni, Zn, Mn etc. (For the eluent, ethylene-diamine/citric acid or ethylene-diamine/tartaric acid is recommended) 2) Analysis of Amine Compounds which have strong hydrophobic group such as R_nN⁺ or R₃N (R : Alkyl group such as Butyl group, Octyl group etc.) (For the eluent, HNO₃ aq./acetonitrile is recommended)
TSKguardcokumn IC-A	Guard Column for IC-Anion-PW and IC-Anion-SW.
TSKguardcolumn IC-C	On cation analysis, eliminate the slight amount of polyvalent cations in the eluent. (Pretreatment of the eluent for IC-Cation and IC-Cation-SW)

4. Configuration and Parts of Columns

Configuration and Parts of Columns are shown in Fig. 1-1 and 1-2.

In Fig. 1-1 configuration and parts of most IC type columns except IC-Anion-PW_{XL} are shown, and in Fig. 1-2 configuration and parts of IC-Anion-PW_{XL} are shown.

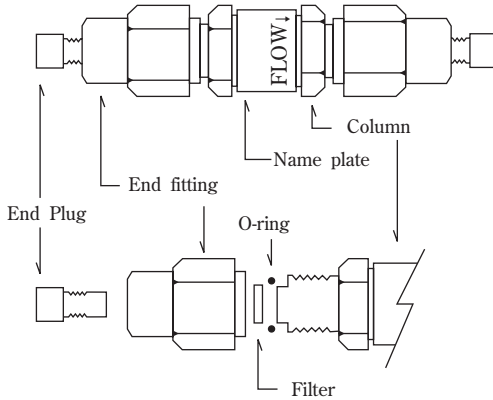


Fig. 1 - 1 Configuration and Parts of Most IC type Columns Except IC - Anion - PW_{XL}.

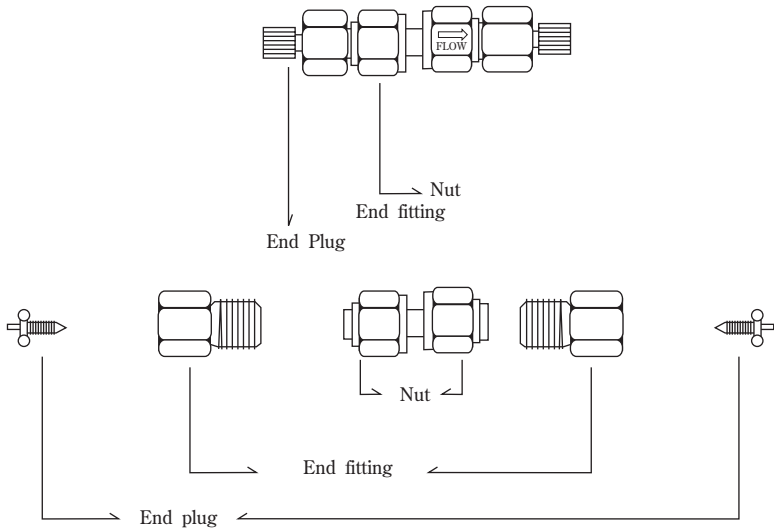


Fig. 1 - 2 Configuration and Parts of IC - Anion - PW_{XL}.

5. Maintenance

5-1 Conditions of Measurement

1) pH Range of Eluent and Sample Solution

Keep pH of eluent and sample solution within the following range.

pH 2.0-12.0.....for TSKgel IC-Anion-PW, IC-Anion-PW_{XL},
IC-Cation and TSKguardcolumn IC-A, IC-C

pH 2.0-7.5..... for TSKgel IC-Anion-SW and IC-Cation-SW

The pH range should be determined from the chemical stability of packing materials.

A chemically modified silica gel like TSKgel IC-Anion-SW and IC-Cation-Sw are easily dissolved by the eluent at pH level above 7.5 and under pH 2.0.

2) Flow Rate

The maximum flow rate for all grades of IC-type columns is under 1.2mL/min(25°C).

For analysis with aqueous solvent, the flow rate of 0.5-1.0mL/min is recommended.

In case the solvent of higher viscosity is used,the flow rate must be set to be smaller.

3) Pressure

Maximum operating pressure should be determined by the withstanding pressures of the column piping and joint.

Use the column at less than 7MPa. Using it at more than 7MPa may cause leakage in the mobile phase or damage to the column.

4) Temperature

The optimal operating temperature for IC-Anion-PW, IC-Anion-PW_{XL}, IC-Cation and TSKguardcolumn IC-A, IC-C is between 0°C and 60°C, and that for IC-Anion-SW, IC-Cation-SW is between 0°C and 45°C.

Below 10°C, apply a lower flow rate to avoid the column deterioration.

5) Ionic Strength

When polyvalent electrolytes have accumulated to the packing material, they must be flushed and stripped from the packing material.

In this case the salt concentration of mobile phase must be under 100mmol/L.

6) Organic Solvents

Organic Solvents miscible with water are often used as a modifier to reduce hydrophobic interaction between sample molecules and packing material.

Allowable concentration of organic solvents for each columns are as follows.

0 - 100 vol. %..... for TSKgel IC-Anion-SW and IC-Cation-SW

below 20 vol.%..... for TSKgel IC-Anion-PW, IC-Anion-PW_{XL}
and TSKguardgel IC-A

below 10 vol.%..... for TSKgel IC-Cation and TSKguardgel IC-C

The organic modifier should be premixed with the aqueous buffer to prevent the precipitation of salts. Exchange of mobile phase between an organic solvent and water containing salts should be carried out intermediately through the substitution with deionized water.

5-2 Preparation of Eluent

1) Reagents

Fresh distilled water, analytical grade of reagents and organic solvents should be used for the eluent.

2) Filtration

The eluent should be filtered through a 0.5 μ m filter before use in order to prevent clogging of the inlet filter and column bed.

3) Degassing

The eluent should be degassed for stable detection.

4) pH Meter

When pH meter is used for pH adjustment, you must pay attention for the contamination of the pH meter by chloride ion.

5) Preventing CO₂ Absorption

If a high-pH eluent is used, reservoir contact with air will spoil reproducibility through absorption of CO₂ which necessarily causes change of pH and ionic strength.

When an eluent of pH higher than 9.0 is used, it is recommended to attach a soda-lime absorbing tube to prevent CO₂ absorption.

5-3 Installation

1) Connections

All columns except IC-Anion-PW_{XL} can be connected with 1/4" -28UNF setscrews to Teflon tube.

For a column of IC-Anion-PW_{XL} can be connected with 1/16" SUS lead pipe of swage lock type.

2) Flow Direction

Use the column in the direction shown by the arrow on the tag attached to the column.

Operating the column with the flow in the reverse direction for a long time will cause degradation of column performance.

3) Prevention of Bubbles

Be careful not to admit any bubbles into the column during its installation or

removal from the equipment. Always remove all bubbles from all pipings before installing the column.

Admitting bubbles into the column will cause degradation of its performance through the occurrence of channeling, etc.

Connect the column as follows:

If solvent leaks from end fitting when the cap on the inlet side column is removed, connect the column to the equipment carefully, as mentioned above, so that no bubbles will be introduced into the column.

If no solvent leaks from the inlet side of the column, connect the outlet side to the equipment and feed solvent through the column in the reverse direction with the feed pump in order to expel the air.

Feed the solvent slowly at less than 1.0mL/min. in this case, rapid pressurization or solvent feeding may cause degradation of column performance.

After confirming bubble - free solvent leakage at the inlet side of the column, arrange the column in the direction of normal flow, and connect the inlet side to the injector.

4) Flow Diagram

Flow diagrams for the analytical purpose are shown in Fig. 2&3.

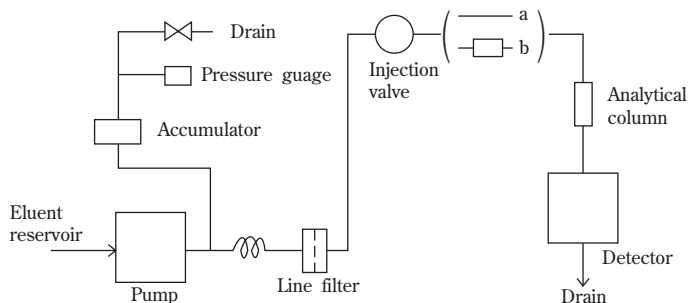


Fig. 2 Flow Diagram for Anions Analysis

a : without a guard column

b : a guard column in use

For IC-Anion-PW_{XL}, use a Filter Assembly (NPR) (Cat. No.14594) attached with the micro-filter less than 0.45 μ m instead of a guard column.

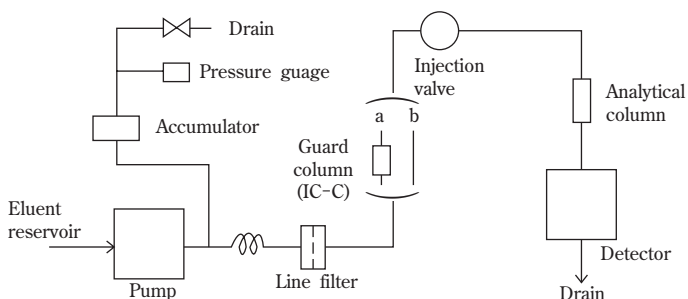


Fig. 3 Flow Diagram for Cations Analysis
 a : for monovalent cations
 b : for polyvalent cations

5-4 Exchange of Solvents (Counter Ions)

The counter ions of each grade are shown in Table 1.

If you want to change the counter ions according to the analytical sample, you can change the counter ions by eluting the solvent used for analysis.

5-5 Preparation of Sample Solution

1) Elimination of Insoluble Matter

Even if nothing can be seen in the sample solution, insoluble matter may be present. So remove the insoluble matter from the sample solution by micro-pore filtration (of eg. 0.45 μ m pore size) but to avoid the contamination by the filter, the removal of the insoluble matter by centrifugation may be recommended.

2) Elimination of Hydrophobic Compounds

To prevent accumulation of hydrophobic compounds in the packing material, it is recommended to eliminate such compounds from the sample by preliminary treatment using commercially available disposable ODS column, etc.

3) Dilution of Sample Solution

In case ionic strength of the sample solution is too high (over 100 ppm per ion) overloading may be occurred and reduce the accuracy of quantitative analysis.

In this case the sample solution must be diluted by the using solvent or deionized water.

4) Concentration of Sample Solution

On the contrary ionic strength of the sample solution is too low, the detection of sample ion becomes difficult. So the sample solution must be concentrated to appropriate level by using "TSK precolumn IC Conc." series. (see Table3)

Table 3 TSKprecolum's Grades and their Properties

Cat. No.	Column Grades	Functional Groups	Applications
08700	TSKprecolum IC-Conc-A	Quaternary Ammonium	Inorganic Anions
08701	TSKprecolum IC-Conc-C1	Sulfonic Group	Alkaline Earth Meta Ions
08702	TSKprecolum IC-Conc-C2	Sulfonic Group	Alkaline Earth Metal Ions Transition Metal Ions

6. Storage of Column

6-1 Protection from Drying and Freezing

Always keep the columns filled with the solvent and tighten the end plugs in order to keep the packing materials from drying and store them at room temperature.

Drying or freezing of column can cause fatal deterioration of column efficiency.

6-2 Storage Conditions

1) Solvent

For IC-Anion-PW, IC-Anion-PW_{XL}, IC-Cation and TSKguardcolumn IC-A, IC-C, the solvent is not necessary to replace for storage.

For IC-Anion-SW and IC-Cation-SW, methanol or acetonitrile should be used as the solvent for long-term storage. (more than one week) However it is not necessary to replace the solvent for routine use.

2) Temperature

Store the column at room temperature. The columns may freeze and their efficiency may degrade if they are left where the temperature is below 0°C.

7. Regeneration of Column

7-1 Clogging of Filter

Insoluble matters in the solvent and the sample solution may cause the clogging of inlet-filter and increases the backpressure of the column and lowers the column efficiency.

If the damage is slight, the column can be regenerated by flushing the column in the reverse flow direction or by replacing the inlet-filter.

7-2 Removal of Polyvalent Electrolytes

Prolonged operation with complex mixture may lead to the gradual accumulation of polyvalent electrolytes compounds.

This is evidenced by changes in chromatography behavior and apparant loss of ion exchange capacity.

Adsorbed material can be stripped from the column by flushing with the following solvents.

Typical cleaning solvents

Buffer containing 100 mmol/L of the salt using for the eluent

.....for TSKgel IC-Anion-PW, IC-Anion-PW_{XL} and IC-Anion

Nitric acid (100 mmol/L)

.....for TSKgel IC-Cation, IC-Cation-SW, and TSKguardcolumn IC-C

7-3 Removal of Hydrophobic Compounds

Flush the packing material in the column with a beffer containing organic solvents.

(Refer the allowable concentration of organic solvents to section 5-16)

The ionic surfactants have hydrophobic section and ionic section, and there is the interaction of static electricity between the packing material and the surfactants.

So you have better to operate in the condition of reducing the interaction.

8. Calculation of the Number of Theoretical Plates and Asymmetry Factor.

The number of theoretical plates (N), the asymmetry factor (As) and their chromatographic conditions are as shown in the Inspection Data.

8-1 Method of Calculating the Number of Theoretical Plates

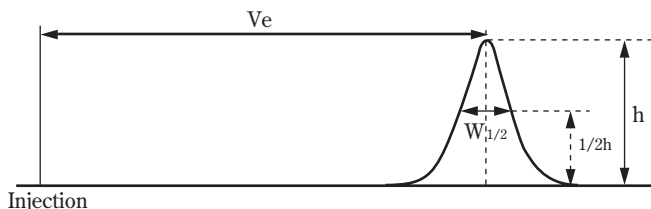


Fig.4 Method of Calculating the Number of Theoretical Plates

The number of theoretical plates of a column (N) is calculated by the half peak width method shown in Fig.4 and the following equation.

$$N = 5.54 (V_e / W_{1/2})^2$$

V_e : Elution volume (min)

$W_{1/2}$: Half width value of peak (min)

h : Peak height

8-2 Method of Calculating the Asymmetry Factor

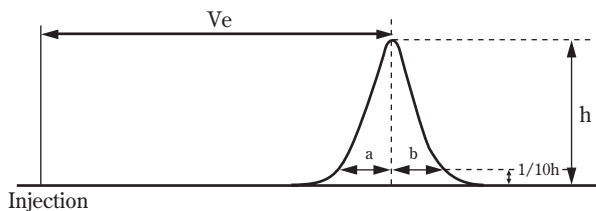


Fig.5 Method of Calculating the Asymmetry Factor

The asymmetry factor of a column (A_s) is calculated by the 1/10 h method.

$$A_s = b/a$$

8-3 Dead Volume

N and A_s should be measured with an instrument of sufficiently small dead volume. indication of a lower number of theoretical plates than the standard value may be caused by a larger dead volume or increased injection volume.

9. Quality Specification and Warranty

9-1 Inspection Data

The results of each inspection are described in the Inspection Data enclosed in the column package.

In the Inspection Data, N is expressed as that per column.

The conditions used in determining the Inspection Data are as follows:

1) Solvent used for the Inspection and Shipping

Table 4 Solvent used for the Inspection and Shipping

Column Grades	Solvent used for the Inspection	Solvent used for Shipping
TSKgel IC-Anion-PW IC-Anion-PW _{XL}	Boric acid buffer (pH 8.5) <Recipe> 1) H ₂ BO ₃360 mg (360 mg) 2) Na ₂ B ₄ O ₇ ·10H ₂ O.....500 mg (575 mg) 3) Glycerine5.0 g (5.0 g) 4) Potassium Gluconate300 mg (350 mg) 5) CH ₃ CN120 mL (40 mL) 6) n-Butyl Alcohol.....30 mL (300 mL) These reagents are diluted by distilled water to 1,000 mL ()show the recipe of IC-Anion-PW _{XL}	Same Solvent used for the Inspection
TSKgel IC-Anion-SW	Tartaric acid buffer (pH 3.2) <Recipe> 2.0 mmol/L Tartaric acid is adjusted to pH 3.2 by 1N KOH	Methanol
TSKgel IC-Cation	2.0 mmol/L HNO ₃ aq.	Same Solvent used for the Inspection
TSKgel IC-Cation-SW	20mmol/L HNO ₃ ag./CH ₃ CN=70/30(Vol./Vol.)	Methanol

2) Samples and their Concentrations for Inspection

Table 5 Samples for Inspection

Column Grades	Samples	Concentrations
TSKgel IC-Anion-PW	SO ₄ ²⁻	10 ppm
TSKgel IC-Anion-PW _{XL}	SO ₄ ²⁻	10 ppm
TSKgel IC-Anion-SW	Cl ⁻	5 ppm
TSKgel IC-Cation	Na ⁺	2 ppm
TSKgel IC-Cation-SW	[(C ₄ H ₉) ₄ N]	500 ppm

3) Conditions of Inspection

- Flow Rate : 1.2mL/min
- Sample Volume : 100 μL
- Temperature : 35°C
- Equipment * : HLC-601 or Equivalet

(*For IC-Anion-PW_{XL} : The following build-up equipment is used.
 Pump : CCPE, Column Oven : CO-8000, Detector : CM-8000)

9-2 Quality Specifications

The shipping specifications of IC columns are shown in Table 6.

Table 6 Shipping Specifications

Cat. No.	Column Grades	N/Column	As
06837	TSKgel IC-Anion-PW	≥ 1,100	0.9 ~ 1.5
14463	TSKgel IC-Anion-PW _{XL}	≥ 1,000	0.8 ~ 1.5
06839	TSKgel IC-Anion-SW	≥ 1,400	0.5 ~ 1.1
07171	TSKgel IC-Carion	≥ 1,400	1.0 ~ 1.8
08055	TSKgel IC-Cation-SW	≥ 2,000	1,2 ~ 2.0

9-3 Warranty:

Im mediately after reciept,check the appearance of the column and test its performance according to section 9-1.

If the guaranteed specifications in Table 6 can not be obtained or the colmn has been damaged during the transportation, contact TOSOH CORP.representative within two weeks.TOSOH CORP. will replance the column at no cost to the purchaser.

No column should be returned to TOSOH without its prior authorization.

The specifications of these columns may change without notice for their improvement.



TOSOH

TOSOH CORPORATION

BIOSCIENCE DIVISION

Shiba-Koen First Bldg.

3-8-2 Shiba, Minato-ku, Tokyo 105-8623, Japan

Phone: +81-3-5427-5180 Fax: +81-3-5427-5220

Web site: <http://www.separations.asia.tosohbioscience.com/>

HPLC database: www2.tosoh.co.jp/hlc/hlcdb.nsf/StartE?OpenForm

TOSOH BIOSCIENCE LLC

3604 Horizon Drive Suite 100,
King of Prussia, PA 19406, USA

Phone: +1-800-366-4875 Fax: +1-610-272-3028

E-mail: info.tbl@tosoh.com

Web site: <http://www.tosohbioscience.com/>

TOSOH BIOSCIENCE GmbH

Zettachring 6, 70567 Stuttgart, Germany

Phone: +49-711-132570 Fax: +49-711-1325789

E-mail: info.tbg@tosoh.com

Web site: <http://www.tosohbioscience.com/>

TOSOH BIOSCIENCE SHANGHAI CO., LTD.

Room 301, Plaza B, No.1289 Yi Shan Road,
Xu Hui District, Shanghai 200233, China

Phone: +86-21-3461-0856 Fax: +86-21-3461-0858

E-mail: info@tosoh.com.cn

Web site: <http://www.separations.asia.tosohbioscience.com/>

TOSOH ASIA PTE. LTD.

63 Market Street #10-03 Singapore 048942

Phone: +65-6226-5106 Fax: +65-6226-5215

E-mail: info.tsas@tosoh.com

Web site: <http://www.separations.asia.tosohbioscience.com/>

HLC, TSK-GEL, TSKgel, TSKgel SuperMultipore, BioAssist, Enantio, PStQuick, Enviropak, TOYOPEARL, ToyoScreen, TOYOPEARL GigaCap, TOYOPEARL MegaCap, TOYOPEARLPAK and TOYOPAK are registered trademarks of TOSOH CORPORATION in Japan and other countries.

This manual may not be reprinted or copied in whole or in part without written consent of TOSOH CORPORATION. The contents of the manual are subject to change without notice.

Printed in Japan