Electrochemical

Accessories

Electrode &



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1. Printed Electrode **Ring Disc Electrode**

Ring Disc Electrode is a printed electrode developed by NTT Advanced Technology Corp. As figure showed below, ring disc shaped electrode is formed



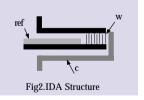
Fig1.Ring Disc Electrode

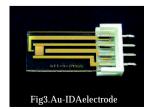
on a glass plate. There is carbon, gold and platinum type of this electrode, and it can be used for radial flow cell electrode. Diameter of electrode disc is 3mm, inner ring diameter 4mm, outer ring diameter 6mm. When it used in radial flow cell, in micro flow it will gain an optimum coulometric electrolysis. At this condition, on the center of electrode disc will occur 100% oxidation (reduction). As special feature, this system can perform qualitative and quantitative analysis simultaneously and also analyze the successive chemical reaction. Using Osmium Gel/HRP (Horse Radish Peroxidase developed by Prof. Adam Heller from Texas Univ.) coated on it, the electrode be able to analyze hydrogen peroxide at zero volt. A flow injection analysis system also performable by modifying electrode with other oxidase to analyze many biochemical compounds.

Cat No.	Description	Qty
002081	Ring disc electrode Au	3
002082	Ring disc electrode Pt	3
002083	Ring disc electrode C	3

IDA (Interdigitated Array) Electrode

Introducing IDA (Interdigitated Array) electrode developed by NTT Lifestyle and Environmental Technology Laboratory. Several articles about determination of micro-substance and observation of its electrochemical behavior using IDA microelectrode have been reported. This microelectrodes made using lithography technology to





form micro pattern upon insulator's plate. Figure3 shows the structure of IDA electrode. Number of finger in one electrodes is 65 pairs. Each electrode works as oxidation or reduction electrode.

Advantage:

- High sensitivity CV measurement
- Very small quantity electrochemical measurement
- Small and integrated
- Fast response

Applications:

- Electrode for liquid chromatography
- Electrode for electrochemical analysis
- Biosensor and chemical sensor
- Chemically modified electrode
- Electrode for Chemical reaction process control
- Conductivity measurement

Cat No.	Description	Width(µm)	Interval(µm)	Length(mm)
002047	IDA Electrode Au	10	5	2
002048	IDA Electrode Pt	10	5	2
002049	IDA Electrode C	10	5	2

CV measurement using Interdigitated Array Electrodes

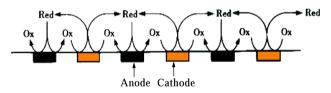


Fig4.Electrochemical redox cycle

IDA Electrode is a pair of band electrode combined and works with each other as a generator electrode and collector electrode, therefore it is possible to make an electrochemical redox cycle upon the electrode as showed in figure. By occurring the redox cycle on electrode increasing electrolysis current to raise measurement sensitivity. In experiment using common electrodes to analyze a small quantity of sample solution, the sample will consumed and exhausted due to electrolysis. However using this Interdigitated array electrode, the oxidationreduction reaction occur repeatedly so the sample solution will not exhausted.

Figure6 show voltammogram of $10\mu L(a)$ (c) and $0.2\mu L(b)$

(d) Ferrocene sample dropped on IDA electrode. The difference between dual mode (a) (b) using both electrode to perform redox cycle and single mode (c) (d) which only one reaction (oxidation or reduction) occur at time can be found clearly. In dual mode (a) (b), the increase of oxidation current on generator electrode will followed by increasing of reduction current on collector electrodes. In (d), response become very small because

consumption of object substance due to electrolysis. Fig5. Redox reaction cycle

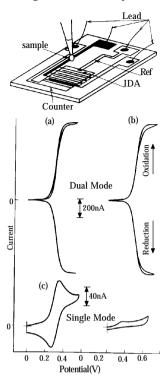
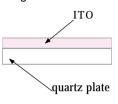


Fig6. Fingerprint analysis by IDA electrode

ITO (Indium Tin Oxide) Transparent Electrode

ITO electrode is a transparent electrode generally used for Spectroelectrochemical measurement. Figure below show an introduction to various products of ITO electrode. ITO layer thickness is 100nm, have about 20Ω electric resistance. ITO transmit light in visible range.



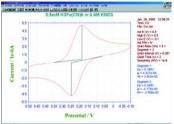
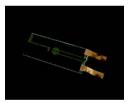


Fig.6 Voltammogram of Ferricyanide by ITO electrode

In addition to ITO vacuum evaporated type flat electrode, we also producing a special order for ring disc shape electrodes, split disc shape electrodes and interdigitated array electrodes.

Fox example, custom made electrode are described here:



Split disc type electrode



Ring disc type electrode

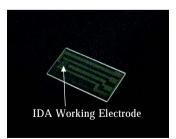


Fig7.ITO electrode optical characteristic

Above electrode is IDA electrode made of ITO material, and you can see its optical appearance.

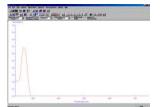


Fig8.ITO spectrum background data

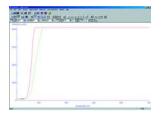


Fig9.Absorption spectrum of ITO electrode and quartz glass (light transmitting intensity)

As figure showed above, ITO show a spectral absorption in ultraviolet range. ITO electrode is a unique transparent electrode material. Custom made electrodes can be made by customer demand.

2.Spectroelectrochemical Cell

Application:

- Electrodes for ion transfer analysis on fluid surface
- Spectroscopic analysis on electrodes surface or near surface
- Spectral absorption analysis of product or intermediate
- To analyze reaction parameter such concentration, diffusion coefficient, life span

Figure10 show the structure of ITO spectroelectrochemical cell and the difference with Pt grid electrodes. The part which transfer light are the ITO electrode and Pt grid electrode part. Measurement performed using fiber optic spectrometer, light source and fiber optic to transmit light with a proper wave length through electrode. Absorption take place to light which pass electrode due to reaction occur on electrode and detected by spectrometer.

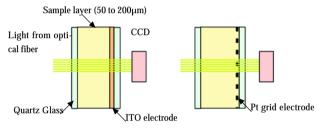


Fig10.ITO spectroelectrochemical cell

Spectroelectrochemical cell Setup



grid working, counter, reference electrode on one quartz plate made of Pt.



Spectroelectrochemical cell and gasket set onto holder



Electrode holder, ITO electrode, gasket, quartz glass cover, screw.



Spectroelectrochemical electrode fix in holder

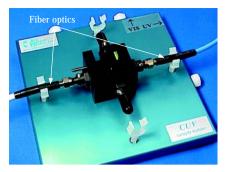


Fig11.Cell set onto Optical cell holder and ready to use for measurement

Connect fiber optics from both side of cell holder with sample is set and one is spectrometer and another is light source. Connect Spectroelectrochemical

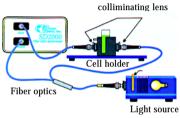
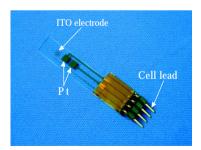


Fig12.Spectroscopic

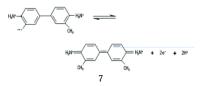
cell terminal to potentiostat, and potential is applied to generate chemical reaction and simultaneously analyzing reaction product using spectrometer.



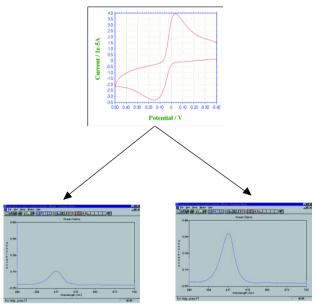
ITO is transparent electrode so it can transmit light. Pt electrodes used as counter and reference electrode.

Fig13.ITO Spectroelectrochemical electrode

3,3'- Dimethylbenzidine (o-Tolidine) used as measurement sample to generate chemical reaction as showed below.



Using thin layer ITO cell, 25µm gasket between ITO electrode and glass cover to perform CV measurement and to observe spectral change take place in reaction simultaneously.



CV voltammogram of 3,3- Dimethylbenzidine

Fig14.Background data

Fig15.Spectral change at oxidation reaction

Absorption intensity change occurs on redox reaction at CV measurement (50mV/s) can be observed clearly.

Depending on samples, when the reaction have a special characteristic in spectral and electrochemical, the reaction mechanism can be studied more detail by analyzing it simultaneously.

Spectroelectrochemical cell accessory

Cat No.	Description
010307	Spectroelectrochemical cell set
010521	USB2000 miniature fiber optic spectrometers
010267	DT-1000 deuterium tungsten halogen light source
010270	CUV-ALL 4 direction cuvette holder
010214	200µm fiber optics (2m)

Custom made cell also available. For instrument details please refer to Fiber Optic spectrometer systems brochure.

3.Electrode for conductivity measurement

Conductivity measurement

Conducting polymer film having a high resistance

(R>10kW), a conductivity measurement commonly performed by simple 2 terminal method. In a film with low resistance (R<10kW), a simple 2 terminal method will be affected by surface leak current. Therefore must provide a guard ring electrode to measure inner current only. Measurement using simple 2 terminal method to measure low resistance sample (high conductivity) will greatly affected by

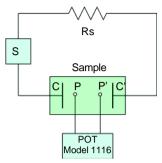


Fig16. Principle figure of resistance measurement using 4 terminal method

- S: constant current electric source
- POT: Potentiometer
- Rs: Standard Variable Resistor
- C,C Current electrode

P,P Potential measuring electrode

the resistance of lead wire used in measurement and joint resistance between lead wire and sample. Measurement using 4 terminal method can be carry out under high resistance sample. A principle of simple 4 terminal method shown at the right figure.

Attach four electrodes on sample, add constant current I from constant current source **S** through current electrode **C** and **C'** into sample. Measure potential difference **U**_{PP'} between two point **P** and **P'** on sample using high input impedance potentiometer (**POT**). At this time, **U**_{PP'} and **Z**_{PP'} (impedance between **P** and **P'**) have a relation as **U**_{PP'} = I**Z**_{PP'}. If distance of potential terminal is **d** and cross section area is **S** then Conductivity **d** is a function as described below.

$$\delta = \frac{\mathrm{Id}}{\mathrm{U}_{\mathrm{pp}}}\mathrm{S}$$

ł

Usually AC current source are used in measurement so this measurement called by AC 4 terminal method also. 4 terminal measurement method using combination of AC source, amplifier and phase detector also applicable.

Conductivity Electrode

For current electrode and potential measuring electrode are fabricated by Pt sputtering on Quartz. Potential terminal are printed with distance as figure below,



It was invented by Osaka National Research Institute.

therefore distance are exchangeable by changing connection terminal. In in-situ measurement of doped conducting polymer generating insulator-metal transition is performed by 2 terminal method on insulator phase low dopant area and with 4 terminal method on

metal phase high dopant area.

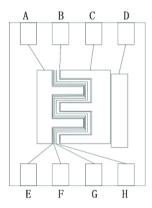


Fig17. Electrode structure

C,D gap is for checking A-H is connecting terminal

Cat No.	Description
010532	Electrode for conductivity measurement

4.ELECTRODES AND ACCESSORIES

Cyclic voltammetry (CV) electrodes

This electrodes is designed for voltammetric use. Small and easy to use for research and measurement in electrochemical field. Electrode is fixed inside PEEK resin which have a good mechanic properties and good solvent resistance including organic solvent. PEEK resin solvent resistance table is showed below. O-ring which fit electrodes body will included in each electrodes to make an easy height adjustment on voltammetry cell.

Polymer	PEEK	Teflon	Polypropylene
Aromatics	Good	Good	Bad
Halogens	Good	Good	Bad
Ketone	Good	Good	Bad
Aldehyde	Good	Good	Bad
Ether	Good	Good	
Amine	Good	Good	
Aliphatic	Good	Good	Good
Organic acids	Good	Good	Good
Inorganic acids	Good	Good	Good
Base	Good	Good	Good

Table 1. Chemical resistance against PEEK polym

Potential Window of Pt, Hg, Carbon electrodes in aqueous solution When aqueous solution with certain pH used as electrolyte and analyze the substances redox activity, it must consider about electrode's hydrogen over voltage, oxygen over voltage and dissolution potential. Figure below is a potential windows figure which show redox potential range

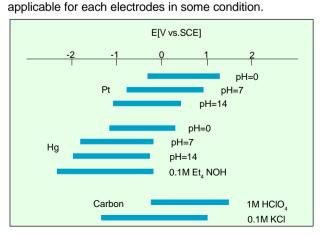


Table 2. Classification of Working Electrodes

Electrode type	Specification	
	Conventional electrodes, having hydrogen	
P t	adsorptive wave, used for H O and oxides	
	detection	
	Conventional electrodes, do not have	
Au	hydrogen adsorption wave, used for thiols	
	detection	
	Chemically stable electrode having large	
GCE	over-potentials of oxygen and hydrogen	
	evolutions	
Ag	For cyanide and sulfide detection	
Carbon nasta	Mixed with enzyme etc. to make modified	
Carbon paste	electrodes	
Nickel	Used to detect amino acids after modified	
NICKEI	chemically	
Pd	Used to study on the adsorption and	
ru	desorption of hydrogen	
PFCE	Highly oriented pyrolytic graphite electrode	
FFCE	surface	

GCE is abbreviation of Glassy carbon electrode, and PFCE is Plastic Formed Carbon Electrode

Fig18.Image of CV working electrodes



Cyclic voltammetry measurement

Linear Sweep Voltammetry (LSV) is a measurement of response current at the potential scan on sample from initial to final potentials at a certain scan rate.

Cyclic voltammetry (CV) is a reversible LSV measurement which scan potential turned to reverse direction after reaching final potential and scan back to initial potential. In CV, reversibility of the products produced in forward scanning can be analyzed during backward scanning. Because of this characteristic CV has been applied widely. If a CV conduct with potential wave form as described below then we can study the substance characteristic as:

- Stability of oxidized and reduced forms.
- Molecular adsorption in redox process.
- Measurements of kinetic rate constants.
- Study of reaction mechanism.
- Reversibility of electrochemical reaction.
- Standard redox potential $E_{p=} = \frac{(E_{pa} + E_{pc})}{2}$
- Electron transfer number $\Delta E = E_{Da} E_{Dc} = \frac{58}{n}$

n: electron transfer number per mole.

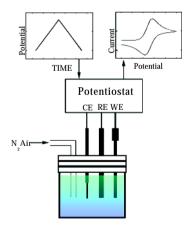


Fig19.Cyclic Voltammetry apparatus

Selection of Working electrode

It is important to choose a proper working electrode for an experiment. Therefore the electrochemical reaction will not interfered by reaction due to the electrode it self and the reaction can be studied in wide potential range. The reaction potential of object substance must be stored inside the potential window of electrode used as showed in potential windows figure in the previous page. Because Pt and Au electrodes have a small hydrogen over voltage and a high oxygen over voltage, it is proper to use as an electrode for organic and inorganic oxidation electrolysis. But because Hg electrodes have high hydrogen over voltage, it is proper to use it for reduction analysis. Conventionally reduction potential of Zinc is not observable covered by hydrogen evolution reaction. But we can observe it clearly using Hg electrode. In measurement of non aqueous solution, hydrogen over voltage and oxygen over voltage are not involved but still must pay attention to the solvent and supporting salt, decomposition potential. And if the non aqueous solution contained water, even in small quantity the potential window become smaller according to the quantity of water.

Figure20 shows i-E curve of $0.5M H_2SO_4$ scanned by Pt electrode for 50 times. 50 cycles scan made Pt electrode activated. On cathode side, an adsorption wave can be observed, and for this reaction need only a constant electric quantity so we can observe the other electrode reaction also in this potential range.

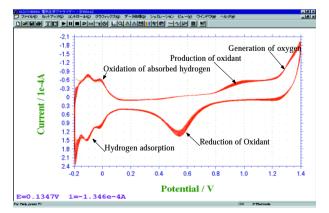


Figure 20. i-E curve of 0.5M H₂SO₄ by Pt electrode.

CV Electrodes and Accessories

Please feel free to contact us to get custom made electrodes

Cat No.	Description	Specif	ication
		O.D.	I.D.
002013	PTE platinum electrode	6 mm	1.6 mm
002313	SPTE platinum electrode	3 mm	1.6 mm
002012	GCE glassy carbon electrode	6 mm	3.0 mm
002411	GCE glassy carbon electrode	6 mm	1.0 mm
002412	GCE glassy carbon electrode	3 mm	1.0 mm
002408	PFCE-3 carbon electrode*	6 mm	3.0 mm
002409	PFCE-1 carbon electrode*	6 mm	1.0 mm
002252	Pyrolytic graphite electrode (Basal)	6 mm	3.0 mm
002253	Pyrolytic graphite electrode (Edge)	6 mm	3.0 mm
002314	SAUE gold electrode	3 mm	1.6 mm
002014	AUE gold electrode	6 mm	1.6 mm
002011	AGE silver electrode	6 mm	1.6 mm
002210	CPE carbon paste electrode	6 mm	3.0 mm
002019	PDE palladium electrode	6 mm	1.6 mm
002319	SPDE palladium electrode	3 mm	1.6 mm
002016	NIE nickel electrode	6 mm	1.5 mm
002017	CUE copper electrode	6 mm	1.6 mm
002018	FEE iron electrode	6 mm	1.5 mm
002250	Platinum mesh electrode	80 mesh	
002251	Gold mesh electrode	100 mesh	I
002223	MCPE carbon paste microelectrode	3 mm	1.6 mm
002005	MPTE platinum microelectrode	4 mm	10 mm
002015	MPTE platinum microelectrode	4 mm	15 mm
002006	MAUE gold microelectrode	4 mm	10 mm
002007	MCE carbon fiber microelectrode	4 mm	7 mm
002002	MCE carbon fiber microelectrode	4 mm	33 mm
002003	MPTE platinum microelectrode	4 mm	25 mm
002004	MAUE gold microelectrode	4 mm	25 mm
002009	MPTE platinum microelectrode	4 mm	100 mm
002010	MAUE gold microelectrode	4 mm	100 mm
002271	MCUE copper microelectrode	4 mm	25 mm
001085	VYCOR [®] glass pipe	3 x 10 cm	
001087	VYCOR [®] glass pipe 10 cm	7 mm	5.9 mm

VYCOR[®] is trademark of corning glass.

Reference electrodes

Reference electrodes can be used in cyclic voltammetry and electrochemical detection in HPLC. We have been classified the reference electrodes as RE series and have a complete line up as for aqueous, non aqueous solvent, calomel.

Application

Cat No.	Description
002058	RE-1C Saturated KCI Ag/AgCI reference electrodes
002057	RE-2C Hg/Hg_SO4, reference electrode. For chlorine less analysis
002056	RE-2B calomel reference electrode
000074	Alkaline mercury oxide reference electrode
002071	Calomel reference electrode for used in alkaline solution

Specification

Cat No.	Length (mm)	O. D. (mm)	liquid junction	component
002058	90	6.0	Ceramics	Ag/AgCl/KCl
002057	115	6.0	Ceramics	Hg/Hg₂SO₄
002056	90	6.0	Ceramics	Calomel
002071	90	6.0	Ceramics	Hg/HgO/ 1M NaOH

Potential of various reference electrodes (V vs. NHE)

NHE(Normal Hydrogen Electrode)	0mV
SCE(Saturated Calomel Electrode)	241mV
SSCE(Sodium Saturate Calomel Electrode)	236mV
Ag/AgCl(Saturated KCl)	199mV
$Hg/Hg_2SO_4(0.5M H_2SO_4)$	615mV

For detailed information refer to Electrochemical Analysis Method (1984) Gihodo, A. Fujishima, M. Aizawa, T. Inoue

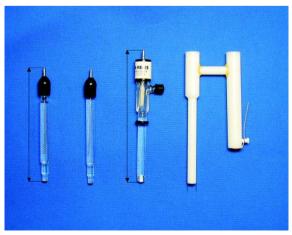


Fig21. Reference electrodes

002057 RE-2C Hg/Hg₂SO₄ reference electrode

Developed for reference electrode with no influence of chlorine ion. RE-2C is made from mercury, mercury sulfate and saturated potassium sulfate solution.

Also available RE-2B calomel electrode, RE-1C Hg/HgCl reference electrode etc. The design are same with RE-2C

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002056 RE-2B calomel reference electrode

Hg_2CI_2 + 2e^- = 2Hg + 2CI^-

E_- = 241mV \text{ vs. NHE } (2.5^\circ)^\circ

002058 RE-1C Hg/HgCl reference electrode (saturated KCI)

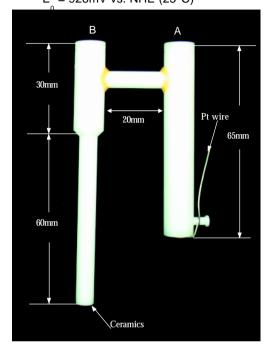
AgCl + e^- = Ag + CI^-

E_0 = 199mV \text{ vs. NHE } (2.5^\circ)^\circ
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002071 Alkaline mercury oxide reference electrode

Reference electrode using Hg/HgO/1M NaOH as a substitute of Hg/ Hg₂Cl₂/KCl in calomel electrode. This electrode can be used in strong basic electrolyte solution.

HgO + H_2O + 2e⁻ = Hg + 2OH⁻ E = 926mV vs. NHE (25°C)



Developed as strong basic resistant electrochemical measure instrument. Using good basic resistance material polyacetal resins. The top of electrode is made as a cap style. At A portion is layered HgO and NaOH solution applied upon it. Pt wire is connected to Hg. On tip of B portion is plugged a ceramic as sample and solution bridging.

Supporting electrolyte

If sample dissolved in organic solvent then supporting electrolyte must be added. On choosing a supporting electrolyte, it must considered about some point below.

- 1. High solubility toward the organic solvent
- 2. A wide potential window
- 3. Inert toward the organic solvent

Some typical supporting electrolyte used : TMAP : Tetra Methyl Ammonium Perchlorate TBAF : Tetrabutyl Ammonium Fluorophosphates

16.ISFET electrode (Cat. No. 001314)

ISFET is an abbreviation of Ion Sensitive Field Effect Transistor. When the sensitive membrane (SiO₂-Si₂N₄) on ISFET gate is exposed on solution, an interface potential (E) occur responding to the ion activity of solution. The sensor is using a Si₂N₄ membrane that responsive to H⁺ to become pH sensor.

When ISFET mV/pH meter used for measurement, since the system using source follower, if drain current (I_d) and voltage between drain and source (V_{dd}) are constant (for example, I_{dc} =100mA, V_{dd} =5V), the potential change in the interface of gate membrane and sample (E) directly displayed as the output voltage (V_{os}) on the meter.

On pH sensing using ISFET, Interface potential E can be expressed as Nernst equation (1). And V_{ns} is dependent to pH. Moreover, to modify gate with various sensitive membrane can create various of potentiometric sensor.

> Where, a_{H} is H⁺ activity, F is Faraday constant, R is gas constant and T is absolute temperature.

(1)

E=const+(RT/nF)Ina_H

Source Drain ISFET

Application of ISFET electrode and references

- 1. Rapid determination of H_O_1
- 2. Na⁺determination²: Na⁺ ion sensor made by coating Na⁺ sensitive substance (Na⁺
- ionophore) on ISFET gate. Application in biosensors³ 3.

Organic compounds can be detected by applying LPL on ISFET gate using glutaraldehyde as bridging material.

H₂COOC-R LPL CH₂OH HÇOOC-R + 3H₂O→ CHOH + 3RCOOH → RCOOH + H₂O → RCOO · +H₃O H₂COOC-R CH_OH Lipid

pH measurement

- 1. Y. Ito, K. Tsutsumi and M. Hirai : Technical Digest of the 7th Sensor symposium, 1988. 157
- 2. Wakita, Yamane, Hiiro : 6thChemical Sensor Symposium, 1987. 85-86
- 3. I. Sato, I. Karube, S. Suzuki and K. Aikawa : Anal. Chim. Acta, 106(1979), 369
- 4. A. Katsube : Materia, Vol34 (1995) No11, P1215-1220.

7.Flow Cell for FIA

The flow cell electrode is made from PEEK. PEEK is hard and have a good resistance to organic solvent, so it can be used in no matter what kind of mobile phase used. Glassy carbon electrode has been applied widely for redox reactions in HPLC. Platinum, gold, Ag, carbon paste and nickel electrode can be used to some special analysis. Thin layer cell was used as electrode in flow injection analysis (FIA) and LC.

Advantage :

- Good stability
- Good performance under acidic, alkaline and organic solvents
- Simple to clean electrode using acetone
- Renewable electrode surface using polishing kit
- Many kinds of electrodes are available
- Easy modification as chemical or bio-sensor

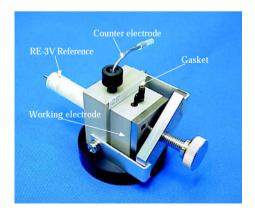
Thin layer flow cell

Cross Flow Cell

In cross flow cell, solvent flow as cross flow on electrode surface, a very basic amperometric cell type. Cross flow cell with flow rate from 1ml/min to 100μ l/min is able to measure electrochemically active substance until 10-15M (femto level).

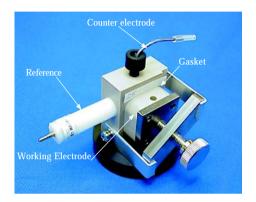
Application :

- 1. Flow injection analysis
- 2. Chemical and biosensor development



Wall jet Flow Cell

Wall jet Flow Cell and Structure of the Working Electrode In cross flow cell, when flow rate are equal or less than 10 ml/min, the detection ability cannot be enhanced. Radial flow cell was specially designed for electrochemical detection under slow flow rate. The detection ability can be enhanced when flow rate are equal or less than 10 µl/min.



Cat No.	Description	size (mm)	Application
001000	GC Electrode	25 x 25	analysis of reductant and
			oxidant
000999	PFCE Electrode	25 x 25	analysis of reductant and
			oxidant
001002	Au Electrode	25 x 25	Analysis of thiols by
			amalgam electrode
001012	Pt Electrode	25 x 25	Analysis of H O and
			oxidant
001008	Ag Electrode	25 x 25	Analysis of cyanide and
			sulfide
001004	CPO Electrode	5 x 25	modified electrodes by
			mixing with enzyme etc.
001009	Ni Electrode	25 x 25	Analysis of amino acids after
			modified chemically
002073	RE-3V Ag/AgCI	48 x 10	for radial and cross flow cells
	Electrode		
001531	1/16" Peek tube 0.25 × 3m, I.D:0.25mm、length:3m		
004130	Dyna seal PEEKPeek resin、connection ferrule, 1/16"		
000201	Tight seal nut Derlin material、connection nut, 1/16"		
000199	Tight seal ferrule Derlin material、connection ferrule, 1/16"		