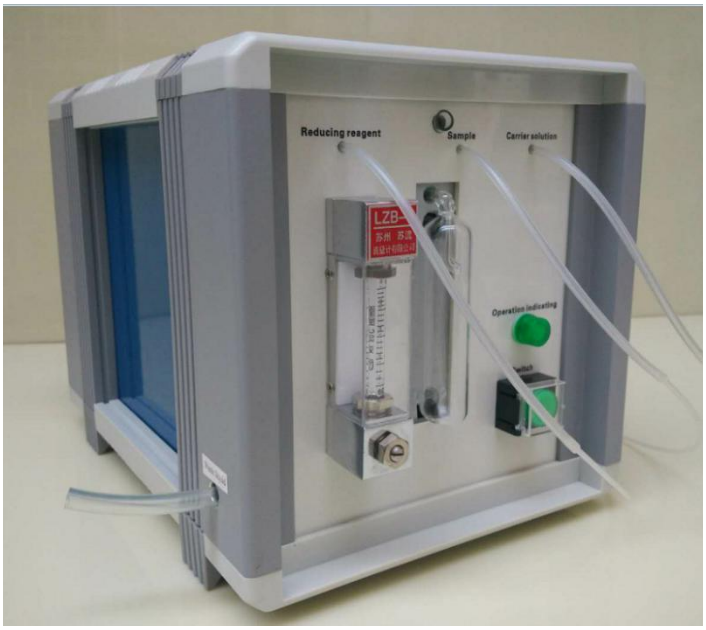
AAH-1 Intermittent Flow Injection Hydride Generator

Operating Instructions

(Please read all instructions completely before operating.)



Content

1.Preface

1. 1 Introduction: This generator is intermittent flow injection hydride generator and it must be used together with atomic absorption photometry. Arsenic, selenium, antimony,bismuth, lead, tin ,cadmium,tellurium, germanium can be determinated by hydride generation atomic absorption photometry and mercury can be determinated by cold atomic absorption photometry.

1. 2 Working condition：Argon was used as the carrier gas，press start button and it will automatically absorb three solution （carrier solution、reducing reagent and sample），emit reading signal when sampling is over, carrier solution impel the sample solution and reducing reagent steady flow, when the three solutions meet in the gas-liquid separator, they will react and emit hydrides. quartz tube atomizer was used for on-line preconcentration of generated hydrides, waste solution is vented from the base of gas-liquid separator automatically.

1.3 Main features：

(1)Unique electronic components control the pneumatic program：automatic sampling with fixed time and capacity、rinse、steady flow reaction，More Contraption, High-performance.

(2)Superior automation: press start buton and the process of sampling, generation, testing and rinse will be finished automatically.

(3)The unique electrothermal quartz absorption tube (It can be used in zeeman atomic absorption spectrometry)：More Contraption，heating quickly, installation easily，steady temperature，longer life , without fuel consume，if only take the quartz tube down and you can change analytical mode quickly.

(4)Excellent analytical performance: the sensitivity of most element is better than 1μg/L/1%A, low detection limit (Take Arsenic for example, the sensitivity is better than 0.18μg/L/1%A).The relative standard deviation is superior to 4% and the test of each time only takes 35 seconds.

(5)Applicability powerfully: All atomic absorption spectrometry can use it.

(6) High reliability: low failure rate, basically no wearing parts.

(7) The weight of hydride generator is only about 2kg, Installation is very convenient.

(8) Three readout modes: you can use peak height(suggested),peak area and continuous reading.

(9) only use little solution (sampling—2-2.5mL,reducing reagent —1-1.5mL and Carrier solution—4.5-5mL)

1. Reducing reagent 2.Sample 3.Carrier solution 4.Operation indicating 5.Switch 6.Separate tube 7.Flowmeter

2. The sequence of installation

2.1 Open packaging,check the amount of goods according to the packing list. If there is any questions, please contact the supplier as soon as possible.

2.2 Install the argon reductor, let argon pipe connect with main gas pipe, and working pressure is 0.2-0.3PMa.

2.3 Set instrument condition and testing parameter according to atomic absorption spectrometry's and hydride generator's operating instructions,about readout mode, peak height readout(readout time is 20s) and peak area readout (readout time is 15s) are both ok.

2.4 Install the hollow cathode lamp, adjust the energy.

2.5 Connect the Joint of Electrothermal quartz tube and install it into the shelves,Make the fixed blade Insert the Burning slit of burner. The position of quartz tube can be adjusted by adjusting the position of burner.

Make the source go through in the centre of quartz tube and adjust the energy at optimal state.

2.6 The output tube of mixed gas which is in the back of hydride generator should connect the electrothermal quartz tube's tubule.

2.7 Electrothermal quartz tube wires connect the output, through 220V power sources. Adjust to 100-110V by regulator. when heating, quartz tube heating wire emit salmon pink light

Note: The type of PE and the type of GBC use 220V voltage directly, power regulator is needless.

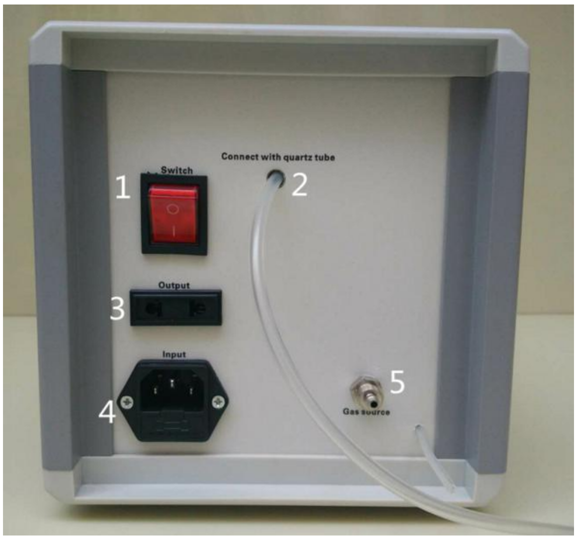
2.8 Open the switch of steel bottle, adjust the output pressure to 0.2-0.3MPa. Open the switch of gas source output, adjust flowmeter to 120-200mL/min。

2.9 Put the tube of waste solution into the waste solution barrel. Make sure that the pipeline is smooth and the tube of waste solution should be on the surface of the waste water. (no need for Water bosh)

2.10 If the mainframe has the automatic reading function, insert automatically reading wire into terminal hole, and connect with remote control socket of automatic reading.

2.11 Insert three plastic tube into carrier solution、reducing reagent and sample respectively, press start button，when sampling is over，the indicator light of relay is quench and the solution begin react and produce much air bubble. This indicates that the instrument is o.k, otherwise it is abnomal

2.12 If above all is OK , you can test the sample referring to the instruction or relevant methods.



1. switch 2.connect with quartz tube 3.output 4.input 5.Gas source

3.Operating condition and operating manual

3.1 Gas source－adjust the pressure of output to 0.2-0.3MPa (setted already)，please use the reductor which we offer. Open the switch of steel bottle and then open the output switch (green button). When testing is over， gas source should be closed.

3.2 The temperature of electrothermal quartz absorption tube－The temperature can be controlled by power supply voltage. The temperature of most hydride element are about 900℃ (salmon pink light). The type of hydride generator use 100-110 voltage which can be adjusted by voltage adjust button.The type of PE and the type of GBC use 220V voltage directly (lower sensitivity).

Mercury can be tested by quartz absorption tube and heating is needless.

3.3 Carrier gas flow rate－optimize the carrier gas flow rate between 100ml/min and 400ml/min. Generally 120－150ml/min is ok.

3.4 Room temperature－The temperature should be higher than 15℃， higher temperature，more sensitivity，conversely reduced.

3.5 The tube of waste solution should be on the surface. Make sure that the pipeline is smooth and there is no need for Water bosh.

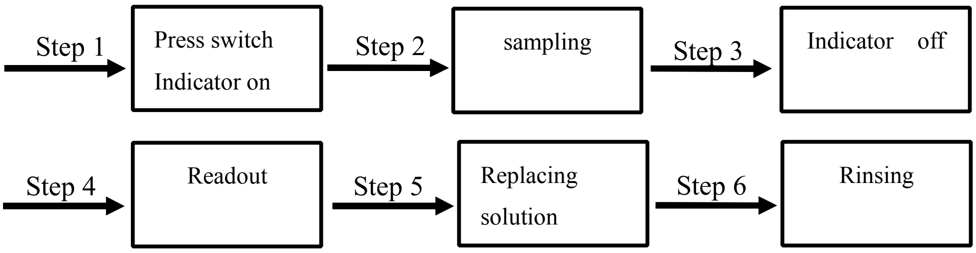
3.6 Pay attention to the valence state of element when testing. When the valence state is uncorrect, there is no sensitivity or the sensitivity is very small. Refer to the operating instructions about the reducing of valence state.

3.7 Acidity is very important when the Hydride elements are testing . When the acidity is uncorrect, it will affect the sensitivity and the stability. Refer to the operating instructions about the acidity of different element.

4. Operating procedure

4.1 This generator only press the start button and replace sample by manual (mainframe needs telecontrol reading), other goes on automatically.

4.2 Test procedure:



4. 3 Sampling：press start button and it will absorb three solutions (carrier solution、sample and reducing reagent) automatically.

4.4 The indicator Light is out - start reading, determination.

4.5 Before Formal determination, in order to clean out the water and air of the pipe, insert sample inhale pipe into the carrier solution and perform twice testing. Before testing, testing blank firstly, the first two data can be deleted. (Testing from low level to high content, rinsing is needless,while testing from high content to low level, rinsing is necessary).

4.6 If testing is over.,rinsing the pipeline, insert three branch pipeline into distilled water and perform three times testing. Then, take out three branch pipeline and do once testing (inhaled air), discharge the waste water of pipe.

4.7 The lowest room temperature should be above 15 ℃, the higher temperature ,the more sensitivity. When the temperature is below 10℃, the testing data is not accurate.

5. Make out the sequence of analytical method

5.1 Firstly, prepare a standard solution that the concentration should be 50 -100 times of the sensitivity (The best absorbency scope is from 0.1 Absto 0.6Abs, high absorbency can cause the bending of curve) and blank solution, which is used to check or confirm:

a.The chemical conditions of occur

b. The conditions of generator

c. The conditions of mainframe

d. The stability of reading

e. Actually sensitivity, (minus the blank reading)

5.2 Confect a series of standard solution, take arsenic for example: 0μg/L、2μg/L、 4 μg/L、6μg/L、8ug/L standard solution and draw standard curve. The range of series standard solution : the highest should be lower the sensitivity of 100－150 times and there must be blank solution.

5.3 Reducing reagent：1.5%KBH_4 in 0.3% KOH(In common condition, it can be used one week).when testing mercury, 0.5%KBH_4 in 0.1% KOH is recommended.

5.4 Carrier solution：1%HCl

5.5 The pretreatment of sample：

a. Dissolved sample refer to related methods. b. Whether the tested element should be prereduction. C. Dilution to certain multiples. d. Reading should be no more than themaximum measurable concentration and meet the requirements of chemical conditions(modified it after measure).

5.6 Check whether there is interferent exist in the sample：
a.Understand the interferent and its quantity of the tested elements from literature material, compared the test sample which coexistence element is known, to determine whether control interference is need or not.

b. Check whether interference is exist by recovery tests： Take 2 copies of sample solution, one add known quantity tested elements, other is not, measure the content and subtraction, calculate the recovery. It is considered no interference when the recovery is in 100±2%. If the demand is higher, there is interference when the recovery is in 100±5% and need to control.

5.7 Interference control
a．Control the interference refer to the method of literature and verifyit by recovery testing.
b. For the unknown interference or can't completely eliminate interference and the recovery is close by adding different amount standard substance, that is to say, "multiplied by interference" is exist and multiplied by a factor can get correct value, standard addition method can be used in this situation. When "add interference" exist, standard addition method cann't eliminate interference.

5.8. Measuring standard sample, verify the correctness of the method.

5.9 Common condition of each element

1. Light source: normal hollow cathode lamp, high-performance hollow cathode lamp is recommend.
2. Take sensitive line as the analytical wavelength.
3. The way of reading：refer to operating instructions, peak height reading is advised.
4. The operating condition of hydride generator：refer to operating instructions
5. Carrier gas: nitrogen or argon
6. When testing by using large concentration solution, the absorbency is larger and the work curve may bend.Using the data to calculate sensitivity, the numerical value of sensitivity will be higher(that is to say, the sensitivity is lower). In order to calculate

sensitivity correctly, we ought to use lower concentration standard solution (The absorbency is about 0.1-0.2) data.

6. Analytical methods for element

6.1.Test for Arsenic

6.1.1 Chemicals and Reagents

All reagents used were of analytical grade.

Borohydride potassium、Potassium hydroxide、Potassium iodide、Ascorbic acid、Hydrochloric acid、100mg/L arsenic standard solutions、Deionized Water.

6.1.2 Apparatus and Materials

Volumetric flask 、Plastic bottle、Pipette、Beaker、Electronic balance、Furnace

6.1.3 Carrier solution : 1% HCl

6.1.4 Reducing reagent: 1.5%KHB_4 in 0.3%KOH

6.1.5 Valence reduction: Take 1mL 100mg/L arsenic standard solutions and join it into 100mL volumetric flask, add 0.8% potassium iodide, pour it into beaker and heated it at a faintly boiling state and add 0.5% ascorbic acid.This standard solution concentration for the stock is As(III)1mg/L. preserve it in dark brown bottle avoiding light, it is usable for half one year and without doing valence reduction.

6.1.6 Standard blank solution : 10% HCl

6.1.7 A series of arsenic standard solution：0μg/L、2μg/L、4μg/L、6μg/L、8μg/L

6.1.8 The valence processing of sample and dilute configuration: adjust the acidity of the dissolved sample to 10% HCl and add 0.8% potassium iodide, pour it into beaker and heated it at a faintly boiling state and add 0.5% ascorbic acid.The stock sample solution should be diluted in the range of the curve when testing.

6.1.9 Sample blank：The same as standard blank.

6.2.Test for lead

6.2.1 Chemicals and Reagents

All reagents used were of analytical grade.

Borohydride potassium、Potassium hydroxide、Hydrochloric acid、Iron hydrogenated potassium、100mg/L lead standard solutions、Deionized Water.

6.2.2 Apparatus and Materials

Volumetric flask 、Plastic bottle、Pipette、Beaker、Electronic balance

6.2.3.Carrier solution : 1% HCl

6.2.4.Reducing reagent: 1.5%KHB_4 in 0.3%KOH

6.2.5 Standard stock solution: Take 1mL 100mg/L lead standard solutions and join it into 100mL volumetric flask and diluted it with 0.5 % to the capacity.This standard solution concentration for the stock is 1mg/L. Preserve it in dark brown bottle avoiding light, it is usable for half one year .

6.2.6 Standard blank solution : 0.5% HCl in 0.8% Iron hydrogenated potassium.

6.2.7 A series of lead standard solution：2.5μg/L、5μg/L、7.5μg/L、10μg/L.

(diluted by 0.5% HCl in 0.8% Iron hydrogenated potassium, and the standard solution can be used for three days)

6.2.8 Sample dilution configuration: adjust the acidity of the dissolved sample to 0.5% HCl and add 0.8%Iron hydrogenated potassium .The stock solution should be diluted in the range of the curve when testing.

6.2.9 Sample blank：The same as standard blank.

6.3.Test for mercury

6.3.1 Chemicals and Reagents

All reagents used were of analytical grade.

Borohydride potassium、Potassium hydroxide、Potassium permanganate、Sulfuric acid、Hydrochloric acid、1000mg/L mercury standard solutions、Deionized Water.

6.3.2 Apparatus and Materials

Volumetric flask、 Plastic bottle、Pipette、Beaker、Electronic balance、Furnace

6.3.3 Carrier solution : 1% HCl

6.3.4 Reducing reagent: 1%KHB_4 in 0.1%KOH

6.3.5 Standard blank solution : 4% H_2SO_4 (add some potassium permanganate and the solution is Slightly purplish)

6.3.6 Standard stock solution： This standard solution concentration for the stock is 1mg/L(diluted by 4% H_2SO_4).

6.3.7A series of lead standard solution：10μg/L、20μg/L、30μg/L、40μg/L (diluted by 4% H_2SO_4).

6.3.8 Sample dilution configuration: adjust the acidity of the dissolved sample to 4% $H_2SO_4$and diluted it in the range of the curve when testing.

6.3.9 Sample blank： The same as standard blank.

6.4.Test for selenium

6.4.1 Chemicals and Reagents

All reagents used were of analytical grade.

Borohydride potassium、Potassium hydroxide、Potassium iodide、Ascorbic acid、Hydrochloric acid、100mg/L arsenic standard solutions、Deionized Water.

6.4.2 Apparatus and Materials

Volumetric flask 、Plastic bottle、Pipette、Beaker、Electronic balance、Furnace

6.4.3.Carrier solution : 1% HCl

6.4.4.Reducing reagent: 1.5%KHB_4 in 0.3%KOH

6.4.5 Standard blank solution : 20% HCl

6.4.6 Valence reduction: Take 10mL 100mg/L selenium standard solutions and 10mL HCl into beaker and heated it at a faintly boiling state and cool it at room temperature.This standard solution concentration for the stock is 50mg/L.

Note: use common glass instruments used, selenium will loss.With polyethylene or polytetrafluoroethylene (opened flask),there is no loss when boiling

6.4.7 Standard stock solution： This standard solution concentration for the stock is 1mg/L (diluted by 20% HCl).

6.4.7 A series of lead standard solution：10μg/L、20μg/L、30μg/L、40μg/L (diluted by 20% HCl).

6.4.8 The valence processing of sample and dilute configuration:pour the 10mL dissolved sample and 10mL HCl into beaker and heated it at a faintly boiling state.The stock sample solution should be diluted in the range of the curve when testing.

6.1.9 Sample blank： The same as standard blank.

6.4.9 Sample blank：The same as standard blank.the reading of absorption should be lower than 0.6A,otherwise the curve will be bend.

Sn

⑴. Wavelength: 286.3nm（normal Lamp) or 224.6 nm(high performance Lamp)

⑵.Carrier Gas Flow Rate: 300mL/min。

⑶. Acidity: 0.5%HCl（V/V）

⑷.Outtake open。

⑸. Linear range:10-80μg/L

⑹.Sensitivity: 0.42μg/L/1%A。

Bi

⑴. Wavelength: 223.0nm。

⑵. Carrier Gas Flow Rate: 100mL/min

⑶. Acidity: 20%HCl(V/V）。

⑷.Outtake: close。

⑸. Linear range: 10-80μg/L

⑹.Sensitivity: 0.42μg/L/1%A。

Te

⑴. Wavelength:: 214.3nm

⑵. Carrier Gas Flow Rate:150ml/min

⑶.Te^{6+} reduction to Te^{4+}**:** Add strong hydrochloric acid into sample or standard solution and boil it about one minute (tellurium can't loss)

⑷. Acidity：20%HCl(V/V)

⑸.Outtake: open。

⑹.Linear range: 10-80μg/L

⑺. Sensitivity: 0.4μg/L/1%A。

Sb

⑴. Wavelength: 217.6nm。

⑵.Carrier Gas Flow Rate: 80-100ml/min。

⑶. Antimony (Ⅴ) reduction to antimony (Ⅲ) : The sensitivity of Antimony(Ⅴ) is lower 2 times than antimony (Ⅲ). Reduction methods is the same as arsenic and it can Pretty instantaneous at room temperatures without heating .

⑷. Acidity：10%HCl(V/V)

⑸.Outtake: Close

⑹.Linear range: 5-70μg/L

⑺.Sensitivity: 0.3μg/L1%A。

Cd

⑴. Wavelength: 228.8nm

⑵. Carrier Gas Flow Rate: 300-400ml/min

⑶.Reducing Agent: 2.5%KBH4 in 0.5%KOH

⑷.Carrier solution：water

⑸.Sample Solution：in $KMnO_4$ with 1.3%HCl (slightly purple)

⑹.Outtake: Close

⑺.Linear range: 2-20μg/L

⑻.Sensitivity: 0.2μg/L/1%A。

Note: The level of mercury is lower than the safe permission level and it won't produce harmful pollution, open the smoke evacuation and ventilation is ok.

7. Routing maintenance and Troubleshooting

1.The cleaning of quartz absorption tube: after long-term use, sedimentary salt which inside the surface of tube make the sensitivity lower. Clean it by hydrofluoric acid(place the absorption tube vertical , use plastic stopper to stopper the mouth and branch pipe and then inject hydrofluoric acid from upper mouth, placed it about 10 minutes and poured out, rinse it with water, dry).

Notice: Do not make acid drops to the electrothermal wire, otherwise insulating layer will be destroyed and short-circuited will occur.

2. When sampling is ok, but there is no absorption. Check the mixed tube of generator and find whether it is obstructed, ensure that they are unobstructed. If there is water, dry it by gas blow.

3. In case of high concentration sample (absorbency > 1A), memory effects will produce, enlarge the carrier gas of the pipeline until the reading of blank is ok(about several minutes or even more than one hour). until the blank reading is ok.

4. If disrelish sensitivity is high:

(1) increase the carrier gas flow

(2) reduce sensitivity by clamping the ventilation tube which behind the instrument, if it still cannot reach requirement, please diluted the solution or contact the supplier.

5. If there is burning fire phenomenon, it indicate that the gas is not pure(oxygen is higher). Clip the ventilation tube tightly and testing again(when in design, the generator automatic joined the air, improve the sensitivity by using the oxygen of air) if it still cannot solve, please change another high purity gas steel bottle.

6. When install and disassemble electrothermal quartz absorption tube, power supply must be cut off . Electrothermal wire and the body of mainframe should not be short circuit.

Note：Different type atomic absorption spectrometry, different performance and also the sensitivity is different. Therefore, doing series standards refer to the specific circumstances and the maximum reading had better not exceed 0.6 Abs, otherwise high concentration may easily cause the bending of curve. The minimum readings should be higher than 0.02 A (deducting the readings of the blank). This generator is for the determination of trace elements, the content is lower or trace, so take special note on removing liquid and the accuracy of volume. Even small error can make great influence on data, so in the process of preparation samples must strictly in accordance with the relevant procedures, clean vessels and confect carefully. If have any question in use, please contact the manufacturer.

Notes： Hydrofluoric acid samples can't be used. If must use, please contact the manufacturer, replace other types of generator. There is 220V voltage in the instrument. Cut off power supply or contact the manufacturer.

8. National Standard of the People's Republic of China

GB/T 16415-1996 Determination of selenium in coal-Hydride generation-atomic absorption method

GB/T 8220.7-1998 Methods for chemical analysis of bismuth---Determination of arsenic content---Ion exchange separation---hydride generation-flame atomic absorption spectrophotometric method

WS/T 129-99 workplace air----Determination of mercury ----Hydride generation atomic absorption spectrometry method

WS/T 130-99 workplace air----Determination of selenium ----Hydride generation atomic absorption spectrometry method

GB/T 12687.3-1990 Chemical analysis of nitrate of rare earth for agriculture-Determination of arsenic content-Hydride generation flame atomic absorption spectrophotometric method

WS/T 109-99 Serum---Determination of selenium---Hydride generation atomic absorption spectrometric method

WS/T 29-1996 Urine---Determination of arsenic---Hydride generation flame absorption spectrometric method

WS/T 47-1996 Urine---Determination of selenium---Hydride generation atomic absorption spectrometric method

National Standard of the People’s Republic of China

Determination of mercury by cold atomic absorption photometry

CJ/T 68-1999 Municipal sewage—Determination of mercury—Cold atomic absorption photometry

DZ/T 0064.26-1993 The test method for groundwater quality-Determination of mercury-Cold atomic absorption spectrophotometry

GB 7468-1987 Water quality-Determination of total mercury-Cold atomic absorption spectrophotometry

GB/T 8914-1988 Hygienic determination method of mercury in air of residential areas--Gold amalgamation-atomicabsorption spectrometric method

GB/T 16012-1995 workplace air----Determination of mercury ----Flameless Atomic absorption spectrophotometric method

WS/T 128-99 workplace air----Determination of mercury ----Reduction gasification -Atomic absorption spectrometry method

CJ/T 98-1999 Municipal domestic refuse-Determination of mercury-Cold atomic absorption spectrophotometric method

GB/T 15555.1-1995 Solid waste-Determination of total mercury-Cold atomic absorption spectrometry

WS/T 26-1996 Urine—Determination of mercury—Cold atomic absorption spectrometric method

9.Packing list

1. AAH-1 Intermittent Flow Injection Hydride Generator
2.Operating Instructions
3.Electrothermal quartz furnace
4.The shelf of quartz tube
5.Argon reductor (nitrogen reductor)
6. Reserved sampling tube
7.Telecontrol automatic reading wiring
8.Screwdriver
9.Two core cable
10.Three core cable
11、Plastic gas source pipe

Delivery date:

Note: if the items are not in accordance with the packing list when open the package, please contact the supplier promptly. The user's special configuration request is not within the scope of the packing list.