Principle and Application

Freeze dryer is a technique that utilizes the principle of sublimation for drying. It makes the dried substance frozen rapidly at low temperatures and then sublimating the frozen water molecules directly into water vapor under suitable vacuum conditions.The substance is always at a low temperature (frozen state) before drying, and ice crystals are evenly distributed in the substance. During the sublimation process, concentration will not occur due to dehydration, and side effects such as foam and oxidation caused by water vapor are avoided. The dry substance is in the form of a dry sponge with multiple pores, and its volume remains basically unchanged. It is highly soluble in water and returns to its original state. To prevent the physical, chemical, and biological denaturation of dry substances to the greatest extent possible.

- For preserving the characteristics of the original substances (e.g. pharmaceutical products or coffee)
- For preserving the original form (e.g. animal preparations, archaeological objects, flowers or books)
- For conditioning the material(e.g. freeze-dried fruit in yoghurt)
- For the sample preparation for chemical or biochemical analysis (e.g. investigation of trace elements in foods, sludge or soil)

Conversion table Vapour pressure above ice

(ice temperature(°C)=pressure (mbar))

°C	mbar	°C	mbar	°C	mbar	°C	mbar
0	6.110	-20	1.030	-40	0.120	-60	0.011
-1	5.620	-21	0.940	-41	0.110	-61	0.009
-2	5.170	-22	0.850	-42	0.100	-62	0.008
-3	4.760	-23	0.770	-43	0.090	-63	0.007
-4	4.370	-24	0.700	-44	0.080	-64	0.006
-5	4.020	-25	0.630	-45	0.070	-65	0.0054
-6	3.690	-26	0.570	-46	0.060	-66	0.0047
-7	3.380	-27	0.520	-47	0.055	-67	0.0041
-8	3.010	-28	0.470	-48	0.050	-68	0.0035
-9	2.840	-29	0.420	-49	0.045	-69	0.003
-10	2.560	-30	0.370	-50	0.040	-70	0.0026
-11	2.380	-31	0.340	-51	0.035	-71	0.0023
-12	2.170	-32	0.310	-52	0.030	-72	0.0019
-13	1.980	-33	0.280	-53	0.025	-73	0.0017
-14	1.810	-34	0.250	-54	0.024	-74	0.0014
-15	1.650	-35	0.220	-55	0.021	-75	0.0012
-16	1.510	-36	0.200	-56	0.018	-76	0.0010
-17	1.370	-37	0.180	-57	0.016	-77	
-18	1.250	-38	0.160	-58	0.014	-78	
-19	1.140	-39	0.140	-59	0.012	-79	

3-6kg
water capture

Item No	BD1-3	BD1-6	BD2-6
24/h water capture capacity	3kg	6kg	6kg
Cold trap temperature	-70℃	-70℃	-90℃
Cold trap volume	5L	10L	10L
Freeze dried chamber	High permeability organic glass/multi manifold external valve/stainless steel chamber		
Sample shelf	φ 200mm stainless steel with 3-5 layers available	• Stainless steel φ 260mm/485mm. 3-10 layers/optional • Electric heating/manual capping/optional	
Vacuum pump	Extreme vacuum $4*10^{-4}$mbar ,with a pumping capacity of 78L~200L/optional pump of various types		
Oil mist filter	Standard configuration		
Vacuum pump connection pipe	Single forming stainless steel flexible pipe DN16 ISO-KF L-1.2m		
Anti-corrosive treatment	Cold trap, condensing coil/all equipped with PTFE anti-corrosion treatment as standard. Freeze-drying organic solvents.		
External valve	6pieces/optional	1-48 pieces/optional	
Eutectic point detection system	Support/Optional		
Data output analysis	Support/Optional		
Power	0.6kw	1.1kw	1.5kw
Size	390*468*448mm	480*498*487mm	

9-12kg
water capture

Tabletop	BD1-9	BD2-9	BD1-12	BD2-12
Floor standing	**BH1-9**	**BH2-9**	**BH1-12**	**BH2-12**
Water Capture capacity	9kg		12kg	
Cold trap temperature	-70 C°	-90°C	-70 C°	-90°C
Cold trap volume	15L		20L	
Freeze dried chamber	High permeability organic glass/multi manifold external valve/stainless steel chamber			
Sample shelf	• Stainless steel φ 260mm/485mm. 3-10 layers/optional • Electric heating/manual capping/optional			
Vacuum pump	Extreme vacuum $4*10^{-4}$mbar, pumping capacity ≥ 200L/can be equipped with various types of pumps			
Oil mist filter	Standard configuration			
Vacuum pump connection pipe	Single forming stainless steel flexible pipe DN16 ISO-KF L-1.5md configuration			
Anti-corrosive treatment	Cold trap, condensing coil/all equipped with PTFE anti-corrosion treatment as standard. Freeze-drying organic solvents			
External valve	1-48 ports/optional			
Eutectic point judgment	Support/Optional			
Data audit trail	Support/Optional			
Power	0.9kw	1.5kw	1kw	1.6KW
Size	390*468*448mm	480*498*487mm		

Square cavity front door opening
4-6kg
water capture

Item No	BF1-6	BF2-6	BF2-9
24/h Water capture capacity	6kg	6kg	9kg
Cold trap temperature	-70°C	-90°C	-90°C
Chamber volume	12L	15L	
Freeze dried chamber	Square cavity/front door opening/door opening angle up to 180 degrees		
Sample shelf	• Stainless steel template partition • Electric heating/optional		
Vacuum pump	Extreme vacuum $4*10^{-4}$mbar, with a suction capacity of 78L~200L, built-in to the host/optional for various types of pumps		
Oil mist filter	Standard configuration		
Vacuum pump connection pipe	Cold trap, condensing coil/all equipped with PTFE anti-corrosion treatment as standard. Freeze-drying organic solvents		
Anti-corrosive treatment	Single forming stainless steel flexible pipe DN16 ISO-KF L-1.2m		
External valve	12 ports/optional	1-48 ports/optional	
Eutectic point detection system	Support/Optional		
Data export analysis	Support/Optional		
Power	0.95kw	1. 65kw	
Size	700x580x1600mm		

One-key Operation
Fast & Easy

Unique Refrigeration
System Design

Low Heat Generation
Stable System

Teflon Treatment
Corrosion & Solvent Resistance

Pirani vacuum sensor
Wide Range & High Precision

Standard ISO-KF
Standard Vacuum Interface

Self Sealing
No need to apply sealing medium

Control System:

5.4" LCD Touch Screen Display

- The display can simultaneously display: all main parameters such as cold trap temperature, vacuum degree, sample temperature, and shelf temperature.
- Schematic diagram of equipment operating components, through graphic display to view the working status of each component of the equipment.
- It has the functions of cold trap precooling and vacuum pump preheating, and the time can be set by the user in the system.
- Display whether the current running status is normal or not by numerical color.
- Built-in saturated vapor pressure & temperature corresponding relationship table, clearly displaying the corresponding relationship.

- One-key operation, the operation is extremely simple, no special professional learning and training is required.
- Thermal electromagnetic over-current and short-circuit protection, making the equipment run more safely and stably.
- Active reminder of equipment maintenance time and vacuum pump replacement lubricating oil time.
- Ambient temperature monitoring, the device automatically reminds the alarm if the ambient temperature is too high.
- Optional eutectic point detection device and endpoint judgment detection system make freeze-drying more scientific.

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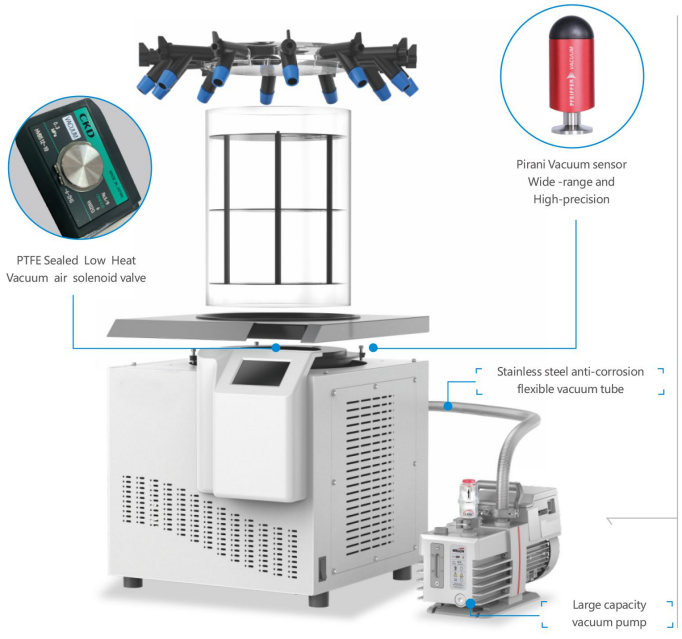
Drying chamber	• The separate drying chamber reduces the probability of damage caused by improper handling of the drying cham ber due to height and weight. The upper cover is anodized aluminum with anti-corrosion treatment. • Highly transparent plexiglass drying chamber, good light and heat radiation conduction. Reserve 6/12 external valve ports. • 316L stainless steel all black Teflon coating drying chamber, suitable for drying organic solvents and various corrosive samples, reserved for 6/12 external ports. • 316L stainless steel vertical multi-port multi-manifold external rack. (The standard configuration is a plexiglass drying chamber, and one of the drying chambers can be optional)
Cold trap and condensing coil	• The cold trap cavity and condensation coil are made of 316L stainless steel. Large opening and channel design, the gas can be quickly captured through the condensing coil. • The cavity and condensing coil are equipped with Teflon anti corrosion coating as standard, which can trap organic solvents and various corrosive solvents. • The condensing coil is placed in the cold trap to increase the condensate catchment area, which can effectively prevent external condensation.
Sample shelves	• 316L stainless steel stackable sample shelves, capable of lyophilizing machine solvents and various corrosive solvents. Each shelf can be placed and taken freely without manual work.The distance and height of each shelf can be adjusted freely. -All black coating, good heat absorption performance. -Special hole for freeze- drying of centrifuge tubes, which is conducive to the drying of samples in centrifuge tube containers. • All-aluminum Teflon surface anti-corrosion treatment electric heating shelf.
Refrigeration system	• Danffos compressor. • Copper plate heat exchanger, the largest area to increase heat dissipation. • Unique refrigeration technology, the temperature of the cold trap can be lowered from ambient temperature to below -80C°within 2 minutes. • Class A temperature sensor with high temperature accuracy and low error. • Multiple detection and alarm system, the refrigeration system pressure is too high or refrigeration failure cantimely alarm and take corresponding measures.



1 Pfeiffer vacuum pump from Germany

2 Welch oil lubrication vacuum pump from Germany

3 Welch chemical anti-corrosion self-cleaning pump from Germany

		1 Pfeiffer vacuum pump from Germany	2 Welch oil lubrication vacuum pump from Germany	3 Welh chemical anti-corrosion Self-cleaning pump from Germany
Vacuum Pump	Type	Two-stage rotary vane oil pump		Chemical anti-corrosion self-cleaning pump
	Limiting vacuum	$4*10^{-3}$mbar		
	Swept volume	84 L	110 L	97~135 L
	Oil mist filter	Standard configuration		
	Interface	International standard vacuum DN 16/25 ISO-KF interface		
Vacuum Sensor	• Anti corrosion Pirani vacuum sensor with automatic temperature compensation control unit, capable of precise detection in low-temperature environments. -Display control range: 1000mbar --- 0.0001mbar -High precision, wide range. -Placed outside the device, it can be easily disassembled for maintenance and storage.			
Vacuum control modulator valve	• Japan CKD High Vacuum Electromagnetic Control Valve: -24V power control, low heat generation, light noise, and long-lasting durability -PTFE sealing material, good sealing performance, chemical corrosion resistance, aging resistance			
Vacuum pump pipe	One time formed stainless steel vacuum tube, permanently non aging, resistant to various types of corrosion.			
Vacuum sealing	The upper and lower end faces are sealed, resistant to aging and corrosion, and there is no need to apply any vacuum sealing grease or sealing medium permanently.			
Oil return prevention measures	• Double oil return prevention measures: -Vacuum pump comes standard with anti return oil device -Vacuum check valve -Active pressure relief system			
Pressure relief and drainage methods	Fully automatic pressure relief and drainage, without manual operation. Completely eliminate damage to samples and sensors caused by unstable manual pressure relief airflow.			

• shelf

• Gland shelf

• Ampoule adapter

• Round bottom lyophilization bottle

• Heated gland shelf

• Wide mouth lyophilization bottle

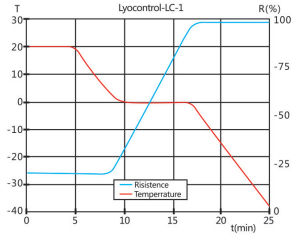
• Vacuum pump lubricant

• Multi manifold

Pilot-scale
Freeze Dryer Series

Process optimization method

Germany Hallan applies Process Analysis Technology (PAT) for process optimization and ensures product quality and process reliability through online monitoring of key control points in the process. The key control points for optimizing the freeze-drying process include: eutectic point of the sample, partition temperature, vacuum degree, resistance, freeze-drying endpoint, and other parameters.



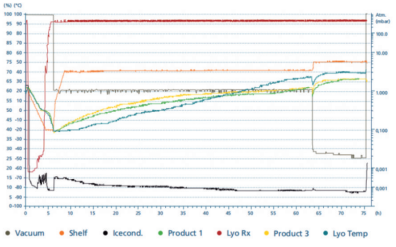
Eutectic poin

Eutectic point is the temperature at which all components of a mixed solution reach the freezing point and begin to form a fixed lattice structure. The temperature of the eutectic point is the upper limit of the sample temperature during the entire freeze-drying process. If the temperature of the sample exceeds the eutectic point, it will cause the disintegration of the crystal structure, and the molten material will break through the surface structure of the crystal, causing shrinkage and splashing of the sample.

Hallan's eutectic point testing device (KM-t system) includes a sample probe that integrates temperature and crystal unit resistance, which can directly detect the impact of different temperature changes on crystal structure, thereby obtaining continuous and accurate eutectic point testing curves.

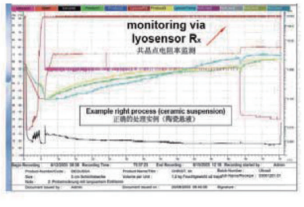
Process curve and process control

The purpose of process control is to divide the entire freeze-drying process into different stages. For example, the main freeze-drying stage is a stage of mass sublimation and solvent removal, while the final freeze-drying stage is a stage of removing residual water and crystal bound water; If necessary, the main freeze-drying and final freeze-drying can be further divided into multiple stages, corresponding to different parameter controls. Record the process curve using the WRL control system and corresponding software. The freeze-drying curve records important data for the entire freeze-drying process and is an important tool for process optimization and batch research. The Hallan freeze-drying curve recording software can record various parameters, including eutectic points, and generate complete experimental reports in PDF and Excel formats.

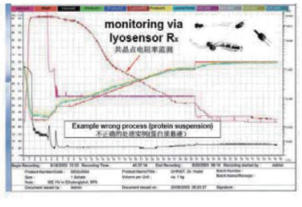


Resistance monitoring

It is crucial to maintain a solid state during the freeze-drying process of the sample, otherwise it will cause structural collapse or shrinkage, affecting its appearance or activity. Based on the characteristics of sample melting and resistance decrease, Hallan has developed resistance monitoring to ensure freeze-drying quality.



Constant resistance, correct process

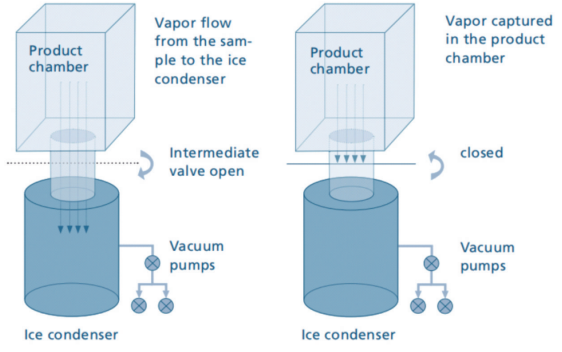


Resistance drop, sample melts

Method for determining the endpoint of freeze-drying

Pressure rise test method

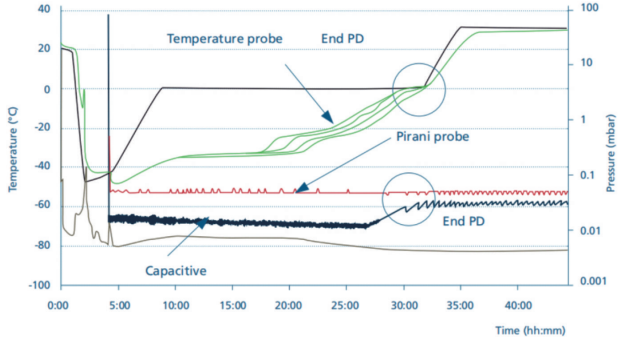
Pressure rise test: A method of determining the degree of dryness of a sample by closing the intermediate valve between the two chambers during the freeze-drying process when the secondary sublimation is approaching its end, based on the rebound of the sample chamber vacuum. The specific range of pressure rebound control needs to be developed through verification and experience or technical support.



Principle of the pressure rise test

Pressure process comparison method

Pressure comparison method: Comparing pressure measurements involves using two different types of vacuum measurement sensors to determine the end of drying. Based on the changes in steam composition, the process monitoring system uses both Pirani and capacitive measuring probes (comparing pressure measurements) to simultaneously measure vacuum. When the water content is relatively high, the difference between the two is significant. As the water content in the sample decreases, the difference between the two will become smaller. The corresponding endpoint can be determined through the recording and comparison functions of the analysis software.



Determining end of drying with the Pirani probe and a capacitive sensor

Pilot-scale Freeze Dryer Series

Hallan pilot-type freeze dryer completely simulates the technical requirements of the industrial freeze dryer, with a compact and reasonable design, a large redundancy range, and conforms to GMP and FDA standards. The ice condensation capacity is 8~24kg, and the WRL controller and liquid temperature control shelf are adopted, so that the process of pre-freezing and freeze-drying can be precisely controlled on the main machine, and the process results are quite comparable with the production machine. In addition to supporting the eutectic point test system, pressure rise and pressure comparison method to determine the freeze-drying end point and freeze-drying curve recording software, hallan pilot-type freeze dryer can also be equipped with engineering WRL control software to directly Graphical computer control the freeze-drying process. It can be configured with H2O2 sterilization and high-pressure steam sterilization or integrated glove box system, which can be used for clean room operation through the wall. The cold trap and freeze-drying chamber are all designed with 316L stainless steel to ensure the cleanliness of the chamber and good ultimate vacuum and vacuum leakage rate to the greatest extent.

hallan pilot type freeze dryer can be used for freeze drying of bacteria, viruses, plasma, serum components, antibodies, serum and vaccines, pharmaceutical products such as chloramphenicol, streptomycin, vitamins, enzymes, plant extracts for biochemical experiments, etc. and research and development.

GMP standard | FDA standard | Eutectic point test | Weighing series | H_2O_2 sterilization | Steam sterilization | Remote control

Technical Parameters:

Model	EX2	EX5	EX8	EX12
Freeze-dried area	0.24㎡	0.48㎡	0.96㎡	1.45㎡
Ice coagulating capacity	4kg	9kg	12kg	18kg
Shelf temperature	-70℃~+80℃			-65℃~+70℃
Cold trap temperature	-70 / -90℃	-70/-90℃	-70℃/-90℃	-70℃/-85℃
Cold trap volume	9L	15L	18L	30L
Chamber type	single	double		
Max number of shelves	2	5	8	12
Shelf size	275*400 mm	275*400 mm	365*465 mm	365*465 mm
Shelf spacing	100mm(adjustable)	100mm(adjustable)	70mm(adjustable)	70mm(adjustable)
Sample quantity (4R Cillin bottle)	558	1395	2630	4230
Shelf heating and cooling method	Thermal conducting silicone oil medium(temperature resistance: -100℃~+600℃)			
Chamber and shelf surface	Atomization and electrolytic surface treatment, no dead corner residue			
Vacuum line	High vacuum stainless steel tube			KF50
External valve port	6/	12/	18/	—
Equipment power	3.8kw	5.5kw	6.5kw	8kw
Equipment size	700*588*1600 mm	1030*700*1630 mm	1030*700*1850 mm	1500*850*1900 mm
Capping mode	Hydraulic system gland device/no gland			Hydraulic pressure
Suitable clean room	suitable			
Vacuum sensor type	PiraniVacuum sensor/capacitive vacuum sensor			
Remote control and alarm	Support 2G/4G/GSM/Wi-Fi			
Terminal judgment function	Pressure contrast method	Pressure test/pressure contrast method		
Freeze dryer front door cover	Stainless steel/Plexiglass			Stainless steel
Inert gas filling	(Optional) Sufficient amount and time for the program to run itself			
User function customization	Can be customized and modified according to user needs			
Sterilization method	H_2O_2/steam sterilization			

Note: "/" optional. Pilot models can be customized according to user needs

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Pilot-scale
Freeze Dryer Series

EX2

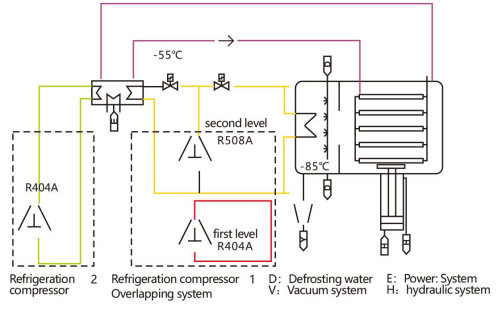
Working chamber

- Double-chamber stainless steel system, optional Teflon anti-corrosion treatment.
- Material: stainless steel 316L
- 1~4 sample temperature probes
- Built-in condenser, high condensation efficiency.
- Plexiglass cold trap window and stainless steel door cover (optional), can observe the ice formation in the cold trap. Applicable to clean room.
- Refrigerant reverse hot gas defrosting.
- Layer temperature uniformity: ±1℃
- Layer flatness≤±0.5mm/m
- Pirani vacuum sensor/Pirani and capacitive composite vacuum sensor

Refrigeration Series

- Dual compressors, Danfoss special compressors, air cooling (water cooling system is optional)
- Refrigeration method: Cascade refrigeration method, high cooling efficiency, good reliability, using CFC-Free environmentally friendly refrigerant.
- Silicone oil conducts heat, with uniform temperature and accuracy up to 0.1°C. Silicone oil has a wide temperature range, up to -100°C~+600°C.
- Shelf refrigeration system and cold trap refrigeration system are completely independent, without any switching, refrigeration temperature is lower and more stable.

EX5/EX8



Pilot-scale Freeze Dryer Series

Vacuum series

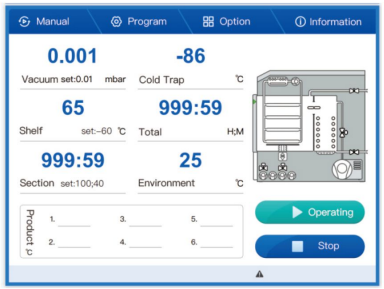
- Vacuum pump:German Pfeiffer anti-corrosion, anti-oil return function vacuum pump. (Vacuum pump can be selected according to user needs)
- Pumping rate: 9m³/h~62m³/h, multiple specifications are optional, the ultimate vacuum can reach 2×10-3mbar, and the oil mist filter is standard.
- The vacuum pipelines are all high-vacuum stainless steel vacuum tubes, which can effectively prevent vacuum leakage and corrosion.
- The double anti-oil return device completely eliminates the damage and pollution caused by the oil return of the vacuum pump pipeline to the equipment and samples.
- Automatic backfill air device, the system has a built-in High efficiency particulate air (HEPA) filter to ensure the complete cleanliness of backfill air.
- The vacuum stabilization system can stabilize the vacuum degree of the freeze-drying process through the micro-pass vacuum control valve to ensure that the samples will not be affected by excessive fluctuations in the vacuum degree.
- Condensed liquid collection and discharge system, automatically completes the collection and discharge of condensed liquid after freeze-drying.

EX5/EX8/EX12

Control System

Characteristic

- Control: Automatic/Manual
- Can store at least 100 independent freeze-drying programs, each program contains 32 program segments (Can be customized according to user needs), each program segment corresponds to a processing stage (pre-freezing, main drying, post-drying).and Include the following segment values:Cycles, shelf temperature, vacuum and safety pressure for the heating unit.
- All the equipment can be recorded in real time and the corresponding freeze-drying curves and data tables can be generated after the end. The records of all data and curves comply with the corresponding regulations and standards.
- It has hierarchical management authority, hierarchical management allocation authority and usage process tracking authority
- Remote System
  - Fully support 2G/4G/GSM/Wi-Fi various connection modes
  - Users can remotely view all data on the device and operate it
  - Technicians can remotely perform maintenance and fault detection on equipment, and can remotely guide and train users
  - Can remotely upgrade and change the system of the equipment



V1 version