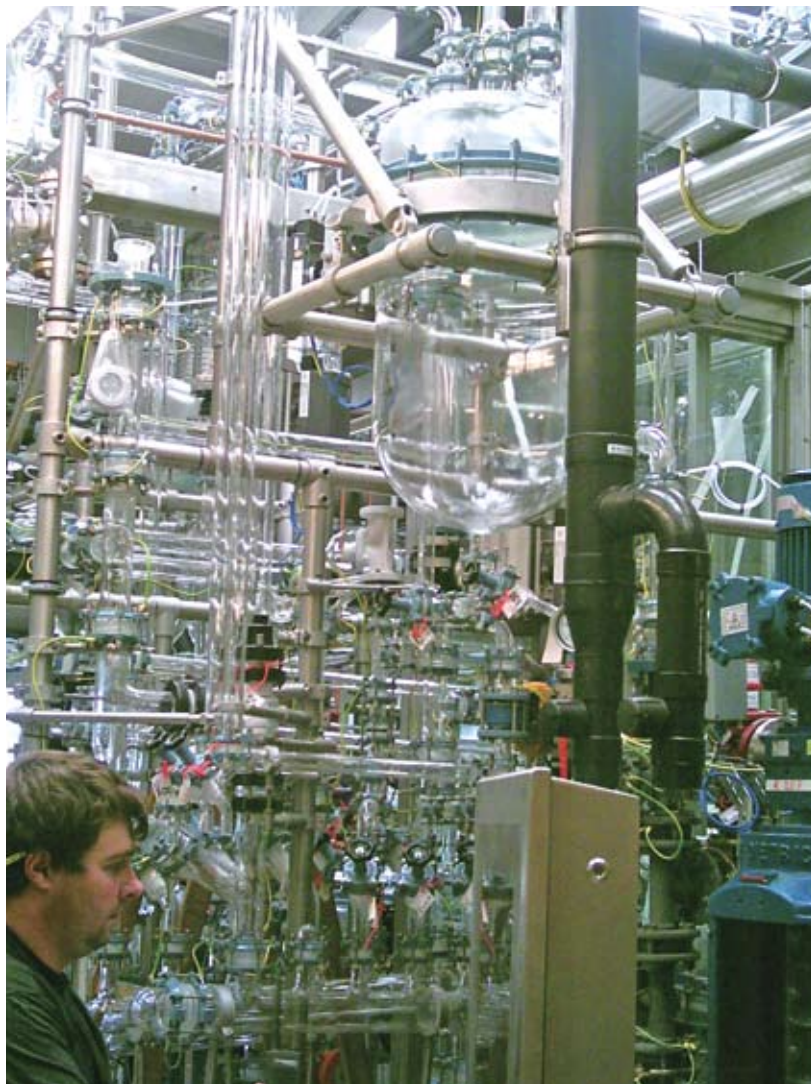


药剂师能看到的情况越多，那么他改进某一流程就越方便
The more the chemist can see, the easier it is for him to influence or change a process



千量实 验室里 的玻璃 工场

玻璃是厂商用来生产数以公斤计的
试验用途产品的理想材料

Glass plants in kilolabs

Glass is the ideal material for plants producing kilograms of product required for trials

当 产品处于从研究实验室阶段过渡到试验工场或生产企业之间阶段时常常会用到千量实验室。顾名思义，千量实验室的主要目的是生产成千数量级的产品。常常有一些

A kilolab is usually employed at some stage between a research laboratory and a pilot plant or production facility. As the name implies, its main purpose is to produce kilo amounts of products. These are often products that have come out of a research laboratory and have not been tested in a stirred vessel; so many parameters are still unknown. Kilolabs also serve the function of developing processes that can be easily duplicated in the pilot plant or in a production facility.



即使两个部分不是完全的对准，每个接头也都能由四颗带有弹簧的螺钉很好的固定
Assembling the join using four screws with springs gives a perfect seal, even if the two pieces are misaligned

刚刚走出研究实验室的产品还没有进行搅拌反应器试验；还有许多的参数未知。千量实验室就是完成一些可以在试验工场或生产企业方便进行重复的研发流程。

收到研究实验室指示的药剂师必须迅速的生产出相当可观数量的产品。他或她需要一套可塑性强的装备，但并不一定是全自动化的，如果是全自动则需要一套本地的人工代用装置。

这样一套装置常常包括一个工作体积在15至250升之间的反应器。一般装有一台涡轮搅拌器，转速在每分钟0至350转即可有效的对内容物进行搅拌。还需要一套加热和冷却设备，工作范围可以根据具体流程设定。

固体物料直接从反应器的开口添加。液体物料既可以通过打开阀门由导管加入也可以由定量给料泵添加，这种泵可以精确的调节流速。

在大多数情况下，设备需要进行蒸馏和分相。一至数台主要冷凝器通常用于蒸馏，另外一台排气冷凝器用于保护真空泵与所有化学物质分开。底部的出口阀门可以将反应器进行分相，或者用分相器将蒸馏物进行分相。这样有时候需要用到一些柱子。

蒸馏物被导入第二个反应器或一个用来收集溶液的简便玻璃容器。可以使用自来水，室内乙二醇系统或一个单独的冷却器对蒸馏器进行冷却。

玻璃是很好的绝缘体，这种属性会随着厚度的增加而增强

The chemist who gets a receipt from the research laboratory has to quickly produce considerable amounts of product. He or she needs a flexible unit, but it should not be fully automated, or if it is it should have a local override.

Such a unit consists usually of a reactor with a working volume of 15 to 250 litres. This is an effective way of stirring the content, usually with a retreat curve impeller and a range of about 0 to 350 revolutions per minute. A heating and cooling unit is also required, whose range depends on the processes.

Solids are directly added through an opening on the reactor. Liquids are either added through the feed vessels by opening valves or by dosing pumps, which can accurately change the flow rates.

In most cases, equipment is needed to distil and separate phases. Distilling is usually done with one or several main condensers in parallel and a vent condenser to protect the vacuum pump from any chemicals. The phases can be separated in the reactor through the bottom outlet valve, or with the distillate through a phase separator. At this point sometimes columns are needed.

The distillate is received in a secondary reactor or a simple glass vessel to collect solvents. Condensers will need cooling, using tap water, an in house glycol system or a separate chiller.

A nitrogen source is needed to overlay the system with the inert gas, and a vacuum source needs to be hooked up for the distillation process. The exhaust gases during the process need to be scrubbed before they can be released into the environment. This can be done with a dedicated scrubber in glass. Filters are sometimes needed for further processes, or other equipment such as centrifuges.

A kilolab can also be used to refine, change or re-evaluate a process. It can also be assigned to a production facility to quickly solve problems showing up during production runs.

Why use glass?

The most obvious critical parameter is chemical resistance. Glass is resistant to all acidic reactions and some of the alkaline reactions. Borosilicate glass is an obvious choice, since units have to be used for a wide range of processes that have already been conducted in a research lab where borosilicate glass has been used. As well as having the advantage of transparency, glass is less costly than any metal.

The more the chemist can see, the easier it is to influence or change a process. In many cases the chemist likes to be able to intervene directly; that is, manually. He or she also needs to record and evaluate what is happening. This means parameters including temperature, pressure, agitator speed and pH.

Because of these advantages, glass is the material of choice for kilolabs.

千量实验室还可以用来提高， 改变或重新评估某个流程

需要配备一台可提供氮气的装置给整个系统供应惰性气体，蒸馏过程还需要吊装一台真空装置。排出的气体在排到环境之前应该进行洁净处理。玻璃的洁净器可以用来洁净空气。此后的流程有时还需要用到滤器，或其他一些诸如离心机的设备。

千量实验室还可以用来提高，改变或重新评估某个流程。它还可以被指派到生产企业以便迅速的解决一些生产进行中出现的问题。

为什么使用玻璃？

最为直接的重要原因是其化学耐受力。玻璃可以耐受所有的酸性反应和部分碱性反应。硼硅酸盐玻璃作为首选的原因是这些装备需要用来试验很多各种各样已经在研究实验室的硼硅酸盐玻璃容器中进行的流程。除了拥有透明的特点，玻璃与金属材料相比还有价格便宜的优势。

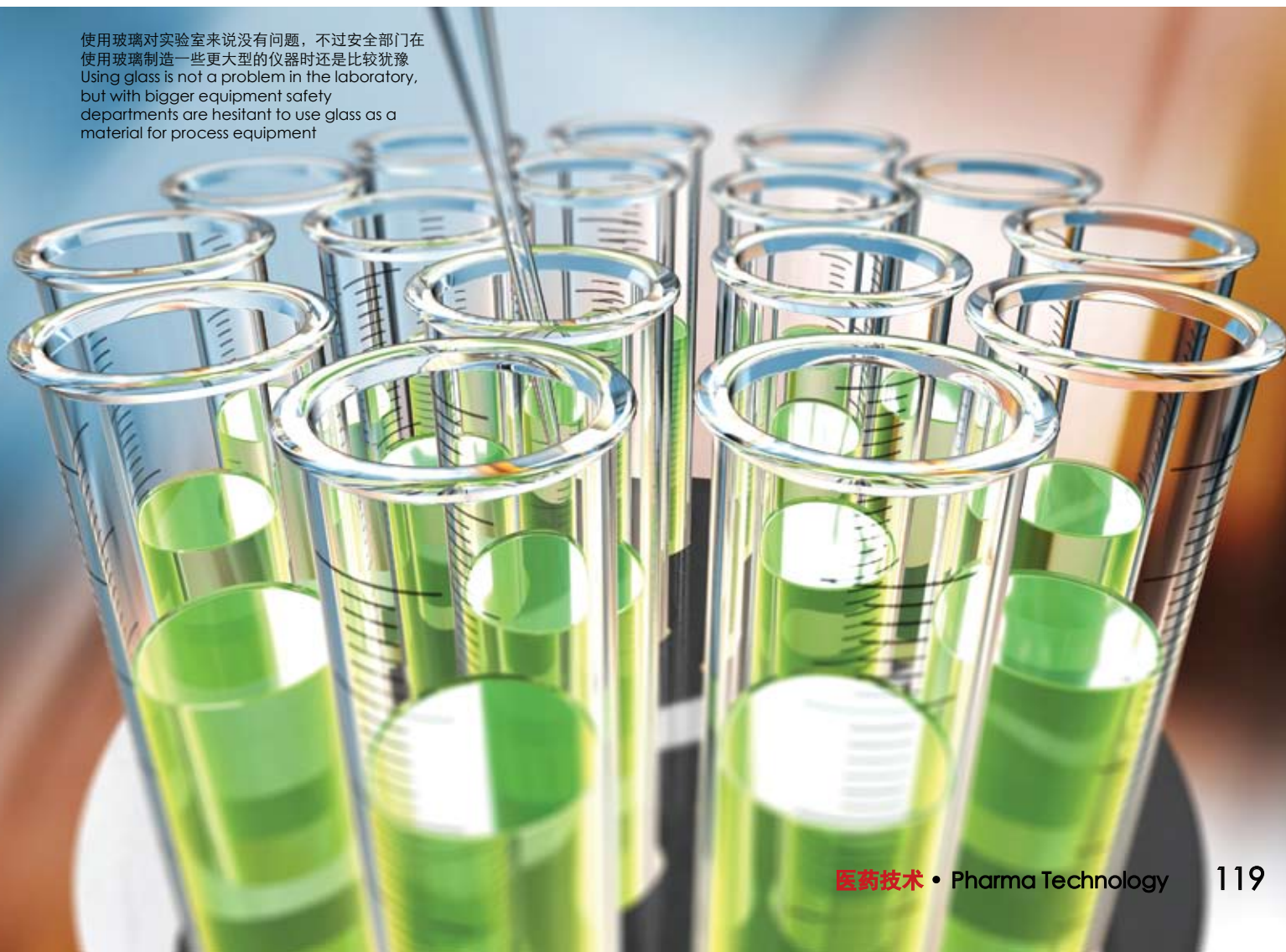
Safety issues

Using glass is not a problem in the laboratory, but with bigger equipment safety departments are hesitant to use glass as a material for process equipment. In Europe there are strict regulations and calculations available to obey before using glass for this purpose. This is why these plants are limited in pressure from full vacuum to a maximum of 0.5 bar, and all units are further protected with rupture discs. Sometimes the units are installed into hoods to further protect the operator, but also to limit the environment which needs to be explosion proof.

Heating and cooling

Another critical parameter in the choice of material is its ability to be heated and cooled. Glass is a good insulator, a property that improves with thickness. That's why the best reactors are glass-lined steel vessels in the bottom section, with glass above the working volume – again for visual reasons. This allows a temperature range of -60°C to +200°C, or as low as - 80°C if

使用玻璃对实验室来说没有问题，不过安全部门在使用玻璃制造一些更大型的仪器时还是比较犹豫
Using glass is not a problem in the laboratory, but with bigger equipment safety departments are hesitant to use glass as a material for process equipment





玻璃可以耐受所有的酸性反应和部分碱性反应
Glass is resistant to all acidic reactions and some of the alkaline reactions

药剂师能看到的情况越多，那么他改进某一流程就越方便。在很多情况下药剂师都希望可以直接干预；也就是说他或她需要手工记录并评估什么情况正在进行中。这里包括一些参数，例如温度，压力，搅拌器速度和pH值。

因为这些优点，玻璃成了千量实验室的理想材料。

安全问题

使用玻璃对实验室来说没有问题，不过安全部门在使用玻璃制造一些更大型的仪器时还是比较犹豫。在欧洲当玻璃用于这一用途时必须遵循一系列严格的规章和计算方法。这就是为什么这些工场压力都被限定在真空至0.5巴之间，所有的装置都进一步受到安全膜的保护。有时候这些设备还会被安装到吊钩上以保护操作工人，不过还是同样限定其周围环境必须是防爆的。

加热和冷却

另外一个选择材料的重要参数是它被加热和冷却的能力。玻璃是良好的绝缘体，这种特性会随厚度的增加而增强。这就是为什么最好的反应器是下部使用玻璃线纹的钢管，工作体积上方配以玻璃——也是为了观察方便。其温度范围在-60℃到+200℃，如果使用特殊玻璃内衬的话可以允许-80℃的低温。对于要求低到-125℃的低

千量实验室防止爆炸注意事项

千量实验室里的药剂师要处理大量的化学药品和溶剂。即使这些东西是被包装着的，但是相同空间内或吊钩上的所有物品都必须是防爆的。一些典型的参数如下：

温度：反应器的温度需要进行控制。依据所选择的加热冷却设施来进行控制。固定的指示可以通过安装一个防爆指示标识解决，而对于那些设定点的变化则需要为设定点提示安装一个防爆面板和调节旋钮。其他需要进行监控的温度还有蒸汽和冷凝物温度，如果每个阶段都安装了一根柱子则需要有另外的温度检测。

压力：尽管反应都是在大气压下进行且蒸馏过程也是尽量在真空状态下进行，但是有时候还是有必要安装一个简单的标尺指示。必须安装一个带有固定传送器和指示器的传感器用于记录压力。

搅拌器速度：无级机械传导或可变频率驱动都可以改变搅拌器每分钟的转速。由于每分钟转速只是一个需要知道但并不重要的测量参数，所以控制工作也不是非常关键。而且在整个流程中它一般也是不会变化的。

其他参数是流速和pH

Protecting a kilolab against explosions

Chemists at kilolabs deal with large amounts of chemicals and solvents. Even if they are contained, everything in the same room or hood needs to be explosion-proof. Typical parameters are:

Temperature: The reactor temperature needs to be controlled. Depending on the heating cooling unit chosen the control might come with such units. The local indication is easily solved with an explosion proof indicating transmitter, whereas the change of set point needs an explosion proof panel with a set point indication and a knob for adjustment. Other temperatures to at least monitor are vapour and condensate temperatures and if there is a column installed each stage needs another temperature measurement.

Pressure: Since the reactions are done at atmospheric pressure and during a distillation as much of a vacuum is drawn as possible sometimes a simple indication with a gauge is sufficient. For a recording of the pressure a sensor is necessary with a local transmitter and indication.

Agitator speed: The changing of the rpm can be done with a stepless mechanical transmission or a variable frequency drive. Control is not so much an issue since rpm is not a critical measurement but should be known. Also it is rarely changed during a process.

Other parameters are Flow rates and pH

硼硅酸盐玻璃作为首选的原因，是这些装备需要用来试验很多各种各样已经在研究实验室的硼硅酸盐玻璃容器中进行的流程

温反应器，需要使用不带玻璃的完全是镍合金制造的导管，外层配以特殊设计的液氮套层。反应器上部玻璃的设计温度范围是-60°C到+200°C。这一限制范围不是因为玻璃的原因而是由于规定使用的垫圈材料为聚四氟乙烯。

真空密封

整个工场的真空密封是另外一个重要参数——不光是为了缩小真空泵必需的体积。由于导入氧气对整个流程是有害的，因此有必要

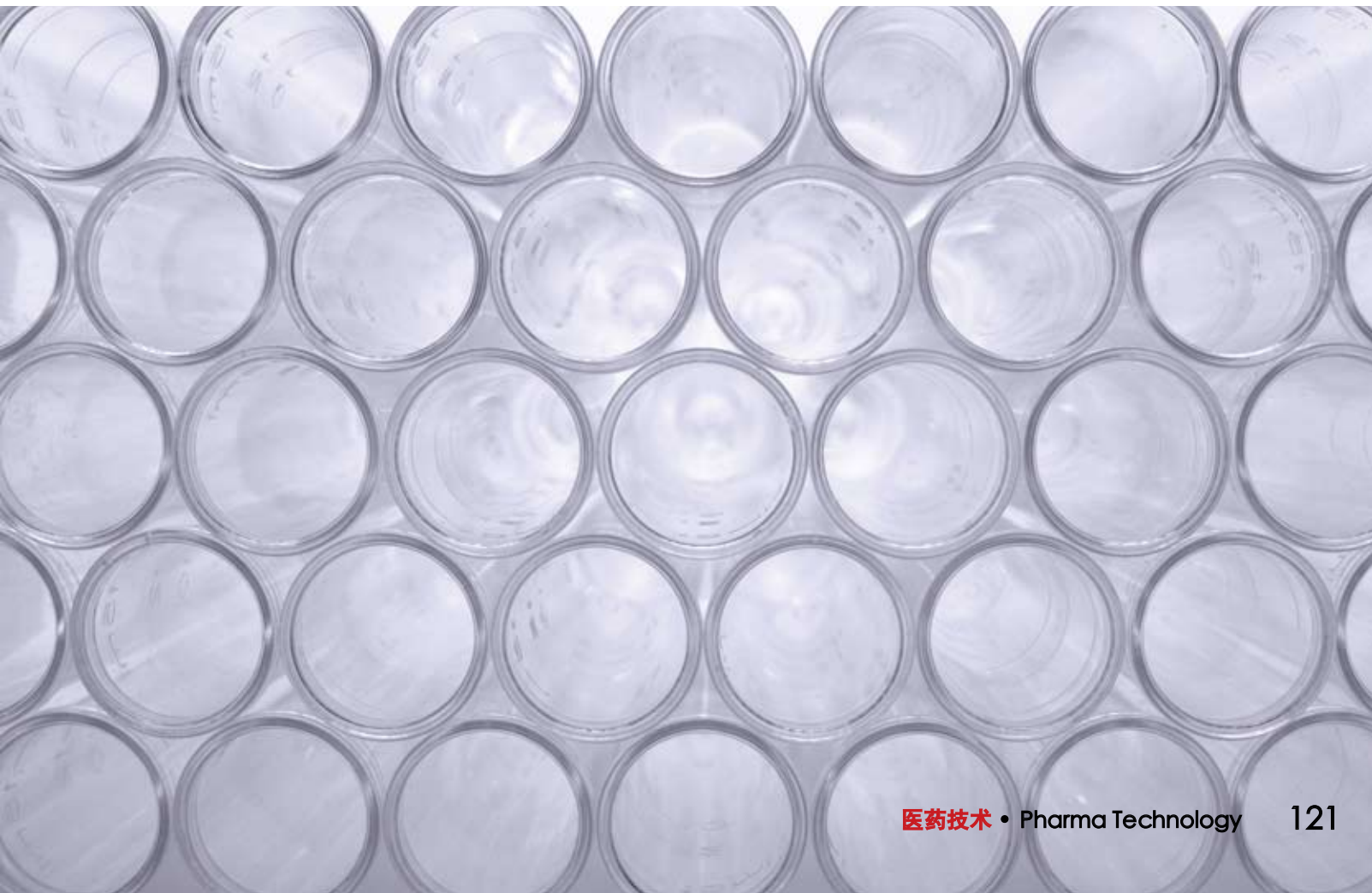
special glass lining is used). For reactions with temperatures down to -125°C, a complete Hastelloy vessel without a glass section is needed, with a specially designed jacket for liquid nitrogen. In all these cases the glass upper section can remain glass, since all separations will occur at somewhat elevated temperatures. The glass upper section is designed for a range of -60°C to +200°C. This limitation is not given by the glass but is dictated by the use of PTFE for the gaskets.

Vacuum tightness

The vacuum tightness of the whole plant is another critical parameter – and not just to minimise the size of vacuum pump required. As introducing oxygen is usually detrimental to the process, it is necessary to be able to create an inert environment. This can be achieved by overlaying nitrogen and minimising the leak rate at each joint or seal.

Büchi discovered a unique way of doing this by designing each joint with a ball and socket arrangement. The socket is a perfectly flame polished sphere, whereas

除了拥有透明的特点，玻璃与金属材料相比还有价格便宜的优势
As well as having the advantage of transparency, glass is less costly than any metal



创造一种惰性的环境。可以通过充入氮气并尽量减小每个接头或封口处的泄漏速度来实现这一目的。

Büchi 发明了一种独特的方式达到了目的——将每个接头设计成一个球配以一个小室的形式。球形的小室内被进行了很好的打磨抛光，而小球带有一个机械加工的聚四氟乙烯垫圈。即使两个部分不是完全的对准，每个接头也都能由四颗带有弹簧的螺钉很好的固定。四根弹簧将可以缓冲任何几度范围内的错配，确保了真空密封性，并且不需要任何额外的柔软性设施。由于聚四氟乙烯可以流动，尤其是在提高温度时，弹簧还要可以缓冲任何的运动。所有的这些因素结合起来可以保证将泄漏速度降到最低并且减少了反应器内氧气的含量。使用这一系统的工场其最小泄漏速度可以达到小于每小时5毫巴。

the ball has a machined PTFE gasket. Assembling the join using four screws with springs gives an almost perfect seal, even if the two pieces are misaligned. The four springs will compensate for any misalignment of several degrees, without compromising vacuum tightness and without requiring any additional flexible elements. Because PTFE flows, especially at elevated temperatures, the springs will also compensate for any movement. All this together will assure a minimum leakage rate and reduce the amount of oxygen introduced into the reactor environment. The leak rate of a medium-sized plant using this system is well below 5 mbar per hour. ■



最好的反应器是下部使用玻璃线纹的钢管，工作体积上方配以玻璃
The best reactors are glass-lined steel vessels in the bottom section, with glass above the working volume

工艺设备 Process Equipment



3D engineering



Large glass overheads

distillation

API's

synthesis

rectification

crystallization

extraction

中试反应设备 Pilot Plants and kilo Lab



minipilot
5 to 10 liters



chemreactors
15 to 250 liters



Nutsche-Filters
10 to 50 liters



Scrubbers
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安全的压力反应 safe pressure reactions

synthesis

**Change the
vessel – not the
entire system!**

polymerization
catalyst testing

hydrogenation
crystallization



**tinyclave
miniclave drive**
10 – 25/50 – 300 ml



Vessel type 1

Borosilicate glass
max. 12 bar



Vessel type 3

stainless steel,
Hastelloy, Tantalum,
Titanium, Zirkonium



picoclave
50 – 300 ml



polyclave
0,25 – 5 l



kiloclave
0,5 – 20 l

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