行政院及所屬各機關出國報告

（出國類別：考察）

mCOC-高性能光電材料技術開發相關事宜

服務機關：(內頁)

出國人員　職　　稱：(內頁)

姓　　名：(內頁)

出國地區：歐洲 (法國，瑞士)

出國期間：89 年 12 月 31 日 -- 90 年 1 月 10 日

報告日期：90 年 4 月 2 日

目　　次

一. 背景說明

茂金屬催化環烯烴共聚物 mCOC (Metallocene based Cyclic Olefin Copolymer)爲新一代高性能之光學塑膠材料，具有優異的光學物性及耐熱性，其密度及吸水率又低，如表一所示，可望取代目前 PC (Poly-Carbonate)及聚甲基丙烯酸甲酯 PMMA (Poly-Methyl Methacrylate)作爲光碟及光學鏡片材料。光學材料又以應用於第三代 HD-DVD 之光碟片基材最被看好，台灣是全世界 CD-R 光碟片之最大生產國，產能佔約 80% ，以 2000 年全世界 44 億片 CD-R 之需求預估，其光碟片基材需求即達 9 萬噸，台灣之需求則爲 7 萬 2 仟噸，因此未來 mCOC 之市場潛力極具爆發性。此外在光學顯示器基板、高頻基板、導光材料及生醫材料等方面如：藥品包裝容器，滅菌－消毒器材，人工水晶體等，與各種耐熱透明保護膜及包裝材等具有甚大應用發展空間。

表一 COC 和 PC, PMMA 的基本物性比較

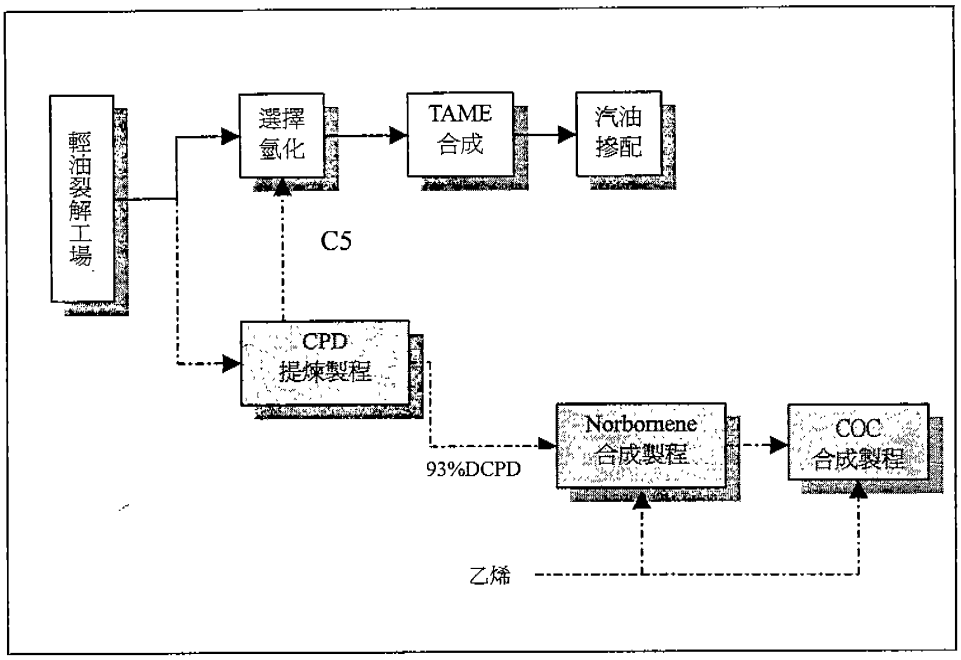
	COC	PC	PMMA
密度/(g cm^{-3})	1.01	1.2	1.2
吸水率/%	<0.01	0.2	0.3
折射率	1.53	1.58	1.49
透光率/%	91	90	92
雙折射率/mm	<25	<60	<20
熱變形溫度/ ℃	123	120-140	80-90
拉伸強度/MPa	64.3	64	70

mCOC 原料來源關聯如圖一所示。mCOC 主要由乙烯和冰片烯(Norbornene)聚合而成，而 Norbornene 則由乙烯和 93%之雙環戊二烯(DCPD)反應而來，DCPD 可由輕裂工場之未加氫之五碳烴爲原料，提煉出五碳烴產品。因此若欲藉中油輕油裂解工場之 C5 原料來開發 mCOC 材料，須涉及三項製程及相關技術之開發：

1.DCPD 之提煉製程

2.Norbornene 合成製程

3.mCOC 合成製程及觸媒開發



圖一 COC 之原料來源關聯圖

中油公司於 84 年及 86 年度委託工研院化工所進行「雙環戊二烯製程技術」研究，即進行 DCPD 之提煉及 Norbornene 合成技術之開發。而工研院化工所 87 年度開始執行經濟部「精準高分子開發與應用四年計畫」科專計畫，中油公司也參與其中「環烯烴共聚物—mCOC 材料開發」，爲未來 mCOC 合成製程開發工作預作準備。

工研院化工所在 DCPD 之提煉製程開發已完成

1. 放大反應與分離系統建立
2. 反應與分離放大驗證
3. 目標產品試製
4. 先導工場單元整合與基本設計
5. 量產製程經濟評估

可望在短期內可完成量產工廠之設計。

但在 Norbornene 合成製程開發方面，工研院化工所目前只在建立實驗室批次試驗系統階段，如果在短期內欲進行 mCOC 之量產，Norbornene 原料之來源便成爲瓶頸。

經查目前有關 Norbornene 合成之專利主要有氣相及液相反應，擁有專利權者有 Dow Chemical Company, Esso Research and Engineering Company, 及 Nippon Oil Company 等。但實際上出售 Norbornene 的有 ATOFINA 公司。工研院化工所即向此公司購買 Norbornene 以合成 mCOC。因此本出國案其中一目的即欲向 ATOFINA 公司洽談購買 mCOC 試驗工場所需之 Norbornene 原料及生產技術，以免將來開發 mCOC 製程時缺乏 Norbornene 原料。

二.拜會 ATOFINA 公司

ATOFINA 公司是 TotalFinaElf 集團之化學品部門，全世界第五大化學品公司。1999 年營業額爲 184 億美金，其主要營業項目包括：

1. 石化及聚合物 (32%)：基本石化原料，聚烯烃，SM-PS-彈性體，VCM + PVC，肥料
2. 特用化學品 (41%) ：樹酯，工業橡膠，表面處理劑，塗料，黏著劑
3. 中間岩原料 (27%) ：氟化物，氯化物，過氧化物，工程塑膠

其銷售地區

1. 歐洲 66%
2. 北美 25%
3. 其他 9%

Norbornene 之生產與 Jean-Pierre POISSON 先生討論。POISSON 先生曾經在生產工場服務，對此工場狀況非常熟悉。

Jean-Pierre POISSON
Division des Polymers Techniques
Product Manager Norbornene-NORSOREX

ATOFINA 公司生產 Norbornene 之工場原屬於 CdF Chimie (煤礦公司)，於 1976 年開始生產。

其工場特點：

1. 具 25 年生產經驗
2. 目前世界上唯一未發生工安事故之工場
3. 不必使用溶劑
4. 工場投資成本低
5. 工場維修費用低

由於 Norbornene 幾 ppm 就有味道，特別向 Jean-Pierre POISSON 請教如何處理。Jean-Pierre POISSON 回覆 ：Norbornene 本身沒有味道但其接觸到氧氣後會形成氧化物而具有刺鼻味道。ATOFINA 公司生產 Norbornene 之工場位於較郊區，且附近之居民皆爲其公司員工，故沒有環保抗爭之問題。但平時工場會派人出來尋查有否 Norbornene 外洩，以免有刺鼻味道之 Norbornene 氧化物漂到遠處而引起環保抗爭。

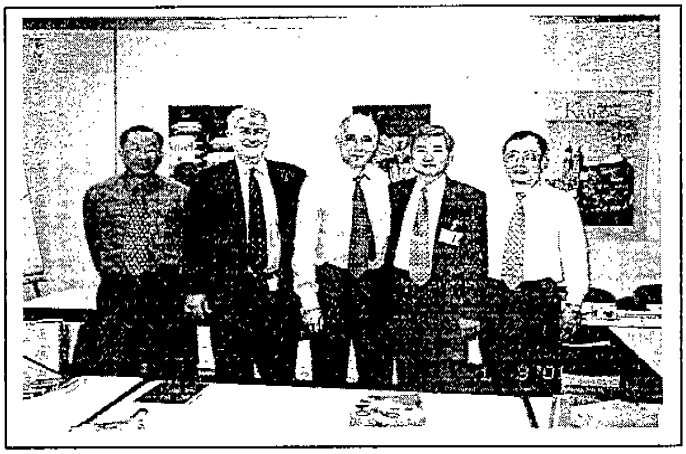
中油公司與 ATOFINA 公司簽有保密合約，故工場之生產狀況無法於此報告詳述。因爲此工場目前爲排定之停工期，且不是在巴黎附近，往返需費許多時間，故 Jean-Pierre POISSON 請提供工場之外觀照片讓我們看。

Norbornene 之銷售與生產技術之出售與 Jean-Michael LARTIQUE-PEYROU 先生討論。

Jean-Michael LARTIQUE-PEYROU

International Division Licensing Department Head Manager

LARTIQUE-PEYROU 表示 ATOFINA 公司願意出售 Norbornene 原料，價格與合約量及下訂單時間有關，可能有四倍之差價。ATOFINA 公司也願意出售 Norbornene 生產技術給中油，但也會出售給其他買主。如中油要求排他，價格一定較貴。



左起胡鑫彬， LARTIQUE-PEYROU， POISSON，洪克銘，洪正宗

三.拜訪 BIAZZI 公司

BIAZZI 公司爲做炸藥之硝基甲苯起家，其特有之技術爲其斧式反應器 (Autoclave Reactor) 之設計，尤其是氣、液反應方面，能將氣體均勻分布並能迅速控制反應器溫度。擁有專利如附件一：

"APPARATUS FOR GAS-LIQUID REACTIONS", US PAT.5,478,535 Dec. 26, 1995.

目前我國有聯勤廠，合益化工，神農製藥等公司使用其斧式反應器。其 user list 如附件二。

BIAZZI 公司斧式反應器之特點如附件三，有：

1. 高質傳
2. 高熱傳 (20 m2/m3)
3. 短滯留時間
4. 氣體均勻分布
5. 緊密性佳
6. 可與熱交換器結合
7. 不會積垢
8. 容易清洗
9. 容易保養維修
10. 觸媒易循環使用
11. 價格不貴

BIAZZI 公司斧式反應器可小至 10 公升，大至 5 萬公升。技術資料如附件四。

在 Biazzi 公司參與討論的主要爲技術人員：

Pierre Marmillod

Director

Marketing and Technical

Dr Jean-Pierre Landert

Head Process Technology

Jean-Jacques Buhrer

Senior Project Manager

Dr Moritz Muhlemann

Process Technology

因 mCOC 之合成爲氣態之乙烯和與液態之冰片烯作用，生成之聚合物其黏度高時若攪拌效果不好時乙烯分散不均勻，易造成生成之產品其品質不佳。故使用 BIAZZI 公司之斧式反應器是正確的選擇。

他們建議可購買 10 公升斧式反應器，如附件三做基礎性探討，並用合作方式進行斧式反應器之放大以便於試驗工場使用。

四. 心得及建議

此次至 ATOFINA 公司洽談向 ATOFINA 公司購買 Norbornene 原料及生產技術之意願。發現其生產技術雖無申請專利，但已有 25 年生產經驗且是目前世界上唯一未發生工安事故之工場。爲值得引進之技術，以解決 mCOC 測試及生產所需原料之問題。

拜會在瑞士日內瓦附近具特有斧式反應器特別技術之 Bizzai 公司，確認其斧式反應器於聚合 mCOC 之適用性，亦爲值得引進之技術。

附件一：BIAZZI 公司反應器專利

US005478535A

United States Patent [19]

Fierz et al.

[11] Patent Number: 5,478,535

[45] Date of Patent: Dec. 26, 1995

[54] APPARATUS FOR GAS - LIQUID REACTIONS

[75] Inventors: **Gérard Fierz**, Lutry, Switzerland; **Peter Forschner**, Hasel, Germany; **Jean-Pierre Landert**, Montreux; **Pierre Marmillod**, La Tour-De-Peilz, both of Switzerland

[73] Assignees: **Biazzi SA**, Chapilly s/Montreux, Switzerland; **Exato GmbH**, Schopfheim, Germany

[21] Appl. No.: **253,863**

[22] Filed: **Jun. 3, 1994**

[30] **Foreign Application Priority Data**

Jul. 8, 1993 [CH] Switzerland 02 054/93

[51] Int. Cl.[6] F28D 11/02

[52] U.S. Cl. 422/205; 422/198; 422/224; 165/109.1; 165/120

[58] Field of Search 422/198, 200, 422/201, 205, 224, 225, 228, 231; 165/109.1, 120; 366/156, 169, 170, 171, 144, 302

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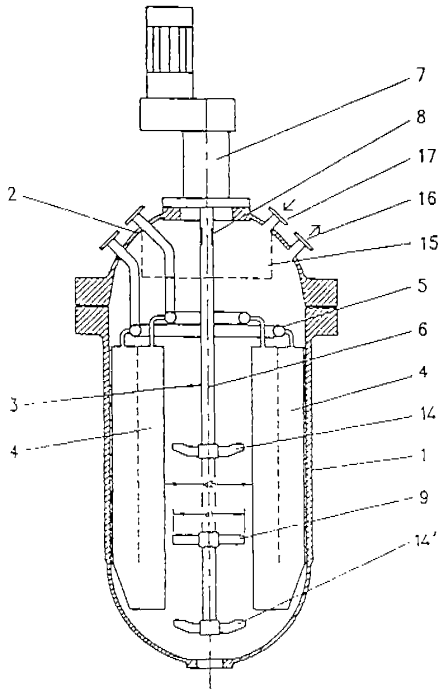
By P. Seichter et al., "Reactor for Catalytic Hydrogenation in the Liquid Phase", Chemical Abstracts, vol. 100, No. 26, Jun. 1984, 212187z, p. 123.

Primary Examiner—Timothy M. McMahon
Attorney, Agent, or Firm—Young & Thompson

[57] ABSTRACT

This apparatus for gas-liquid reactions includes a vessel (1) which is gas-tight and capable of withstanding pressure, a rotatory agitator device (3) to provide for the gas-liquid contact and a heat exchanger device. This heat exchanger device is provided as an assembly of plates (4) positioned approximately vertically around the agitator device, in such a manner as to form an angle of 0 to 70° with respect to the radius, in the direction of rotation of the agitator device, and a heat exchanger fluid circulates inside these plates.

7 Claims, 3 Drawing Sheets



附件二：BIAZZI 公司反應器之 User List

BIAZZI REFERENCE LIST HYDROGENATION & OTHER REACTION GAS/LIQUID PLANTS

No.	*Year of sale*	*Product*	*Capacity m^3 / bar*	*Customer*	*Execution*	*Country*
1.	1991	Pilot	0.1 / 50	BIAZZI SA	standard	Italy
2.	1992	Pharmaceutical	3 / 40	DIPHARMA S.p.A.	cGMP	Italy
3.	1992	Toluidines	2 / 50	NAMHAE CHEMICALS	standard	S. Korea
4.	1993	Confidential	3.5 / 50	DEGUSSA	standard	Belgium
5.	1993	Confidential	12 100	HOECHST CLARIANT	standard	Brazil
6.	1994	Pharmaceutical	12.5 / 45	HOFFMANN LA ROCHE / OMV CHEMIE	cGMP	Austria
7.	1994	Aromatic amines	5 / 50	KOTHARI S.& C.	standard	India
8.	1994	Pilot	0.1 100	BIAZZI SA	standard	Italy
9.	1995	Pharmaceutical	2 / 40	SIEGFRIED AG	cGMP	Switzerland
10.	1996	Confidential	6.5 / 50	KYOWA YUKA	standard	Japan
11.	1996	Pilot	0.1 / 100	AKZO NOBEL	standard	Netherlands
12.	1996	Confidential	40 / 20	FLEXSYS (MONSANTO/AKZO)	standard	Belgium
13.	1996	Confidential	11 / 90	DIXIE CHEMICALS	standard	USA
14.	1996	Confidential	5 / 7	Confidential	standard	Japan
15.	1997	Confidential	10 / 60	ELF ATOCHEM	standard	France
16.	1997	Pharmaceutical	2 50	OMNICHEM	cGMP	Belgium
17.	1997	Confidential	11 / 35	Confidential	standard	Germany
18.	1997	Furfuryl alcohol	1 45	INDUNOR	standard	Argentina
19.	1997	Confidential	15 conf.	Confidential	standard	Japan
20.	1998	Pharmaceutical	0.1 / 50	SANOFI	cGMP	France
21.	1998	Pharmaceutical	2 10	COURTAULDS CHEMICAL	cGMP	Switzerland
22.	1998	Confidential	2x14 / 110	FORMOSAN UNION Chem.Corp.	standard	Taiwan R.O.C.
23.	1998	Pharmaceutical	2 40	ISOCHEM	cGMP	France
24.	1999	Pharmaceutical	0.5 / 100	Confidential	cGMP	Germany
25.	1999	Pharmaceutical	0.4 60	SIGMA-ALDRICH	cGMP	England
26.	2000	Pharmaceutical	2 40	NOVOCHEM 2000	cGMP	Spain
27.	2000	Pharmaceutical	2.6 / 20	SCINOPHARM	cGMP	Taiwan R.O.C
28.	2000	Pharmaceutical	0.1 / 20	RHODIA	cGMP	France
29	2000	Confidential	20..'..28	SINOPEC	standard	China

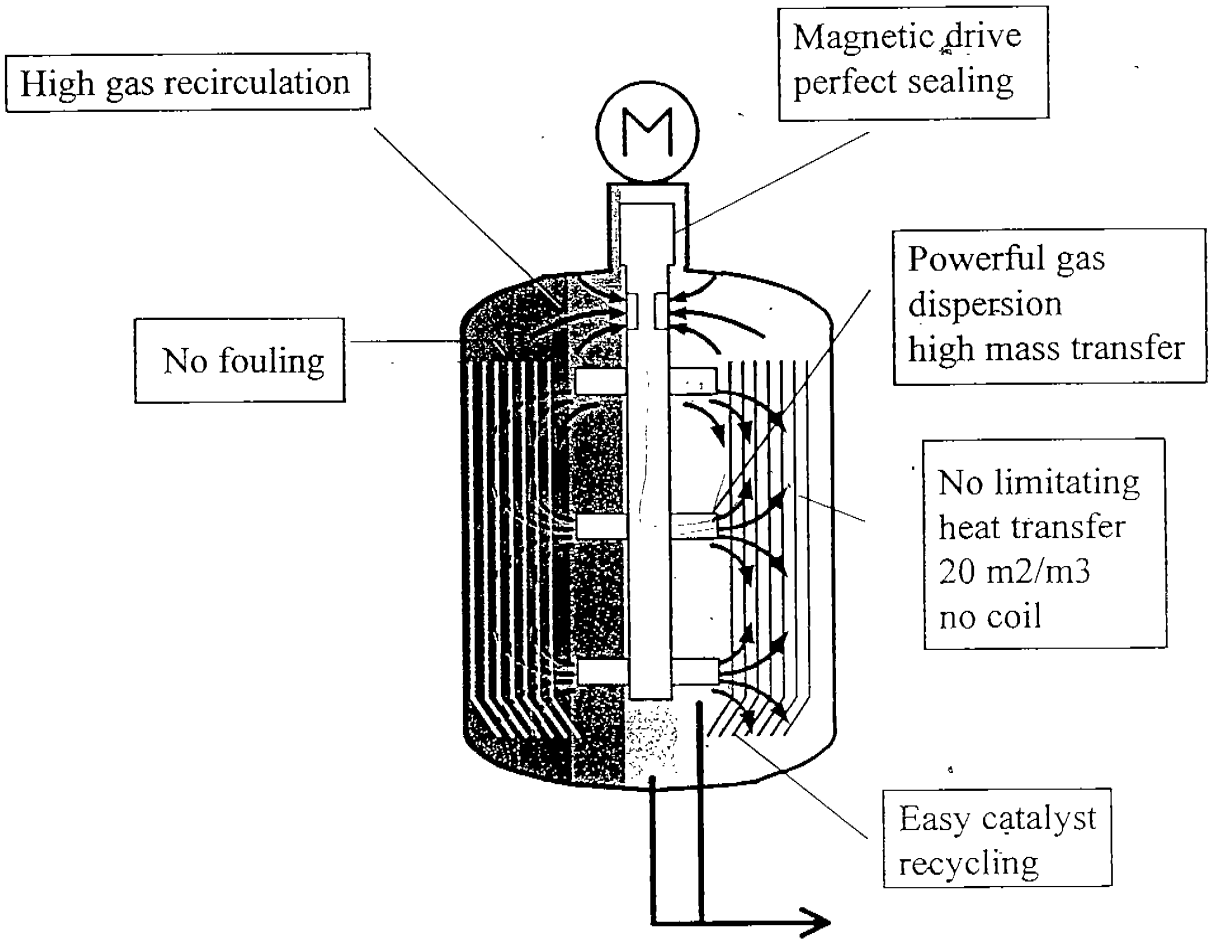
附件三：BIAZZI 公司反應器特點

NEW HYDROGENATION REACTOR

IDEAL INDUSTRIAL REACTOR:

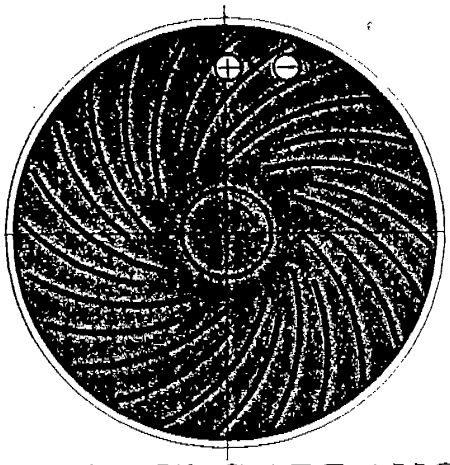
- No mass transfer problem.
- Powerful heating/cooling system + accurate temperature control.
- Low deactivation rate and easy recycling of the catalyst.
- No sealing problems. Low maintenance required.

THE BIAZZI HYDROGENATION REACTOR



(PATENTED)

THE BIAZZI HYDROGENATION REACTOR



ORIGINAL PLATES ARRANGEMENT

PARTICULAR REACTOR FEATURES

- **Permanent oversaturation** of gas
- **Homogeneous conditions**
- Quasi **isothermal** conditions
- **Excellent heat transfer** (20m2/m3)
- Good cooling / heating even with stirrer out of order
- **Minimum fouling**

ADVANTAGES OF BIAZZI REACTOR

- **High mass transfer** (ideal reactor)
- **High heat transfer** ($20 m^2/m^3$ reactor volume)
- **Short batch time** due to powerful heating and cooling
- Reaction usually **without solvent** (no heat removal limitation)
- Homogenous repartition of gas (**permanent oversaturation of catalyst**)
- **Perfect tightness** = high safety (due to magnetic drive)

ADVANTAGES OF BIAZZI REACTOR

- **Integrated heat-exchanger** (particularly useful in urgent situations)
- Practically **no fouling**
- **Easy cleaning**
- **GMP Execution**
- **Easy maintenance** (drive in gas space)
- **Easy catalyst recycling** (settling in reactor)
- **Compact execution** (more economical)
- Possible **external recirculation**/purification of gas

附件四：BIAZZI 公司反應器之技術資料

7/3/4 1D

TECHNICAL NOTE

concerning the

BIAZZI

KILO-LAB UNIT (10 L)

for

HYDROGENATIONS

and other

GAS-LIQUID REACTIONS

CHAILLY/Montreux, December 2000

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FOREWORD

The present document is a technical description of the ***BIAZZI Kilo-lab unit (10 litres)*** for hydrogenations and gas-liquid reactions under pressure.

BIAZZI also offers standardised pilot plant units with capacities up to 500 litres. Beyond this limit, BIAZZI offers industrial scale plants (from 1 to 50 m^3) which can be specified according to the customer's requests.

The information contained in this proposal shall be considered as STRICTLY CONFIDENTIAL and shall not be disclosed to third party without BIAZZI's written authorisation.

1. INTRODUCTION

BIAZZI has extensive know-how and considerable experience in the design, construction and operation of industrial catalytic hydrogenation plants.

BIAZZI is able to supply a complete and integrated know-how and engineering package as well as the related process equipment required for catalytic hydrogenations and other gas-liquid reaction plants, starting from the feedstock up to the corresponding final purified product and including, when required, the related waste water treatment facility.

Our R&D facilities are fully equipped with laboratory and pilot plant hydrogenation units and this also enables us to develop manufacturing process for new products. With the BIAZZI system, all required guarantees, in particular scaling up of the process to industrial manufacture, could be provided.

As a new development, BIAZZI, thanks to its experience accumulated in the last 10 years and its patented system, can now offer to you a ***Kilo-lab unit***, giving you the possibility :

⇨ To develop processes *in your laboratory*, which you are ensured to scale up exactly to industrial production scale by means of a BIAZZI system.

⇨ To allow sample batch production.

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This ***BIAZZI Kilo-lab unit*** is designed for liquid phase catalytic hydrogenations, as well as for other gas-liquid batchwise reactions, including :

- Hydrogenation of double/triple bonds to saturated derivatives
- Hydrogenation of aldehydes and ketones to alcohols
- Hydrogenation of nitro-compounds to amines
- Hydrogenation of nitriles to amines
- Aromatic ring saturations
- Hydrogenolyses
- Carbonylations
- Oxidations
- Alkylations
- Aminations
- Others

2. BIAZZI PATENTED SYSTEM

2.1 Catalytic liquid phase hydrogenation

The hydrogenation reaction is characterised by being highly exothermic and having high mass transfer resistance. An efficient hydrogenation system must therefore incorporate the following features :

- **High rate of heat removal** so as to control accurately the temperature profile of the reaction in accordance with the process requirements and to ensure an optimally short batch time, i.e. high productivity, by avoiding the limitations resulting from poor heat transfer.

- **High rate of gas-liquid transfer** so as to overcome the mass transfer barrier and to ensure a high reaction rate even at a low catalyst concentration.

- **Efficient agitation** providing a high shearing effect so as to renew continuously the product layer surrounding the catalyst, which is critical for obtaining a high reaction rate.

BIAZZI, to overcome these difficulties, has developed a high performance hydrogenation reactor of unique design.

The ***BIAZZI Kilo-lab*** **reactor** is a scale-down of this BIAZZI reactor, which consists of a pressure vessel fitted with both a patented agitation system and patented cooling/heating elements of high surface area.

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The agitation system has a hollow shaft turbine agitator, which provides the required gas-liquid mass transfer, high shearing effects, a high internal recirculation rate of hydrogen and liquid circulation in an optimal way. A very high mass transfer rate (k_La value) is achieved.

The agitator is driven by means of a practically maintenance free magnetic coupling system which avoids the well-known sealing problems associated with high-pressure equipment.

The cooling/heating elements are not of the pipe bundle type, which would impair the required agitation, nor coils on which deposits of catalyst or product could form during draining operation.

Rather, they are of a special vertical plate type and are arranged in such a way that proper circulation as well as high turbulence of the reactor contents is ensured.

2.2 Advantages of the BIAZZI reactor

The advantages presented by the BIAZZI hydrogenation reactor design are numerous :

- **High productivity** due to the fast reaction rate which is achievable, and which allows the batch time to be shortened to the minimum.
- **Low catalyst consumption** thanks to the high mass transfer rate obtained and the permanent hydrogen over-saturation of the catalyst, which allows the operating cost to be minimised.
- **High quality of the products** thanks to the high conversion and yields obtained, as well as the excellent selectivity during the hydrogenation reaction.
- **Near isothermal conditions** because the heat transfer elements are located in the reaction zone and not externally as in certain other systems which attempt to give a high performance. This is particularly important for heat sensitive products, the quality of which could be affected by local overheating.
- **Absence of mechanical seals** and an external circulation pump which require delicate and time consuming maintenance in high-pressure duty. Most of the difficulties met with medium and high-pressure hydrogenators arise from the sealing system of the drives.
- **Use of a magnetic drive**, the design of which is such that practically no imaginable failure could cause a leakage into environment.
- **Excellent batch to batch reproducibility of results**, because all operations are automatically controlled.
- **Reliable and guaranteed scaling-up** from the Kilo laboratory plant to production plant thanks to the know-how that has been developed.
- **Safety of operation** thanks to the reliable control and interlocking systems used, as well as the safety devices incorporated in the plant.

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- **Full cGMP execution.** Many industrial cGMP plants have been delivered to the pharmaceutical industry

3. GENERAL

3.1 Description of the BIAZZI kilo-lab unit

The ***BIAZZI Kilo-lab unit***, executed **according to cGMP** standards, consists essentially of :

- **One gas-liquid BIAZZI pressure reactor,** which provides high mass transfer and high heat transfer thanks to its unique patented design.
- **One temperature control unit**, consisting of one pump, one expansion vessel, and two heat exchangers (1 electrical heater, and 1 chiller with compressor, cooled with water). The heat transfer medium is thermal oil.
- **One gas (usually hydrogen) feed control system.**
- **One catalyst filtration unit.**
- **One vent gas scrubber unit**

The schematic diagram *"kilo-lab unit (10L)"* shows the arrangement of the above equipment.

3.2 Capacity

The capacity of the ***BIAZZI kilo-lab*** **unit** is :

- Useful capacity : 4 to 8 litres
- Operating pressure : 0 to 90 bar
- Operating temperature : -10 to 250°C max.

3.3 Instruments and controls

The standard hydrogenation reactor is equipped with the necessary instruments for an efficient process control. Additional instrumentation is also available to meet special requirements (monitoring, data acquisition, semi-automatic operation).

The unit is offered with the possibility of operating :

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⇨ **Manually :**

The operator performs all operations. The temperature inside the reactor is controlled automatically at its set point by means of temperature control system, which includes a heating and chilling unit.

Or :

⇨ **With semi-automatic control :**

The unit (installed on a explosion proof area) is controlled from a local control board installed in a safe area.

A basic interlocking of the remote controlled valves and safety system is assured by means of a PLC system.

Some automatic or semi-automatic sequences are pre- defined, as well as the order in which they may occur.

The temperature of the reactor contents as well as the pressurisation and venting of the autoclave are automatically controlled.

Other operations are manually performed by means of local push-buttons on the control panel.

A RS 232 plug enables to connect the PLC to a data acquisition system not included in the standard delivery.

This system provides the flexibility required for operation of a multipurpose pilot plant, without impairing its reliability and safety.

3.4 Codes of construction

The reactor is designed according to PED (European code) or ASME code section VIII Div.1. Other codes also possible on request.

3.5 Space requirement

3.5.1 BIAZZI Kilo-lab unit

The complete unit consisting of reactor, the agitation system, the gas scrubber, the catalyst filtration unit, all piping and valving and the instruments, wired to a junction box, are mounted on a stainless steel structure (skid) that can be ***installed in a standard walk-in fume hood***.

This structure includes a pneumatic lifting device, which allows the reactor vessel to be raised and lowered easily. The command for this device is local (start and stop buttons on the structure).

The space required by the ***BIAZZI Kilo-lab unit*** is approximately :

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1'200 mm x 550 mm x 1'500 mm (W x L x H)

3.5.2 Equipment installed outside the walk-in fume hood

- The control panel : approx. 600 mm x 500 mm x 1'300 mm (W x L x H)
- The temperature control unit : approx. 460 mm x 550 mm x 1'340 mm (W x L x H)

3.6 Hazard classification

Equipments installed inside the walk-in fume hood :

All equipment, instruments and electrical connections installed on the skid in the walk-in fume hood are designated as **explosion proof** (Eex).

Equipments installed outside the walk-in fume hood :

The control panel and the temperature control unit are not designated "Eex," but all signals between these equipment items and the junction box installed on the skid are Eex-i.

4. BRIEF DESCRIPTION OF PLANT AND PROCESS

4.1 Operation of the plant

A typical operation of the plant can be briefly described as follows :

The catalyst suspension, previously prepared by the operator, is transferred manually by mean of a funnel into the hydrogenation reactor (previously filled with material).

The temperature of the reactor contents is brought to the set point by circulating thermal fluid from the temperature control unit through the reactor heat exchanger plates.

Once the set temperature is established, hydrogen feed to the reactor is initiated. Heat removal is carried out by the circulation of thermal fluid, which is cooled by passage through the cooling heat exchanger in the temperature control unit. When the reaction is completed, the contents of the reactor can be cooled down to the appropriate temperature, prior the filtration of the catalyst. The contents of reactor are transferred by means of nitrogen pressure into the catalyst filter.

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4.2 Temperature control unit

The internal heat transfer elements in the reactor are connected to a temperature control unit using thermal oil and providing accurate temperature control, particularly during the transition phases of the reaction during which heating has to be switched to cooling and vice versa.

This compact unit has a very fast heating and cooling rate and provides a cooling capacity of higher than **4'000 W at 100°C**.

The choice of the model of temperature control unit and of thermal oil depends on the of the temperature range of the reaction.

4.3 Hydrogen feed control system

The hydrogen feed is controlled :

Either :

⇨ *Manually by the operator* :

A flow indicator (flow range : 0 .... to 40 NL/min.), a manual needle valve and the local manometer allow the H_2 feed and the pressure in the reactor to be controlled. According to the process requirements, the operator will open or close the manual needle valve and adjust the H_2 feeding pressure on the compressed hydrogen bottle.

Or :

⇨ *Automatically by the PLC :*

In this case, the system consists of a thermal mass flow meter (execution Eex i b IIC T4), an Ex-control valve (execution Eex E II T4) and a pressure transmitter (0 ... 100 bar). The operation of the control valve is made via the PLC. The flow range is 0 .... to 40 NL/min.

4.4 Catalyst filtration

The hydrogenated reaction mixture containing the catalyst in suspension is fed by nitrogen pressure (3 - 4 bar) into the filtration unit. The filter is basically formed of a vertical cylindrical body and filtration is carried out by passage through a horizontally placed filter medium (this can be e.g. paper, sintered metal...). The hydrogenated product is discharged via the bottom valve and the solid is recovered on the filter medium. This operation is manual.

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4.5 Gas scrubber

All the gases discharged by venting during loading/unloading operations of the reactor are sent to a gas scrubber where they undergo a washing with water (or solvent) before release. Possible further gas treatment is under the Customer's responsibility.

5. MATERIAL OF CONSTRUCTION - MAINTENANCE

All the parts in contact with the product are of stainless steel 316 L or equivalent or in Hastelloy (on request).

Gaskets are made out of PTFE or Kalrez (chosen to avoid frequent replacements).

All equipment is mounted on a stainless steel structure. The hydrogenation reactor is submitted to the tests defined in the official construction code. The whole unit (including the temperature control unit) is then workshop assembled, pre-mounted and tested before shipment.

Thanks to the proven and reliable technology provided (the magnetic drive of the agitation system, in particular), the maintenance of the plant is limited to routine checks and occasional replacement of valving gaskets.

6. PRODUCTS AND UTILITIES

6.1 Products and catalyst

Materials processed would be expected to include :

- **Liquid products**, such as :
  - organics,
  - aqueous solutions with pH > 4 (except for Hastelloy execution),
  - mixtures, suspensions, etc.
- **Hydrogen**, with a supply pressure of 100 barg maximum (reduction of hydrogen pressure to 100 barg, if necessary, is in the customer's scope of supply). The use of other gases is also possible. Please contact BIAZZI.
- **Catalyst** of different types, such as :

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- nickel or cobalt powder (Raney type),
- nickel on support (silica or others),
- noble metal on support such as activated carbon,
- others as required.

6.2 Utilities required

- **Process water** (for vent gas scrubber)
- **Cooling water** (to cool the compressor included in the temperature control unit)
- **Nitrogen** (for pressurisation of the reactor and filtration) :
  - max. pressure : 7 barg
  - min. pressure : 3 barg
- **Electric power** :
  - 3 x 400/230 V ; 50 Hz or 60 Hz

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Enclosures :
** BIAZZI kilolab unit Kilolab 10L "schematic diagram"*
** Kilolab_cote.pdf*

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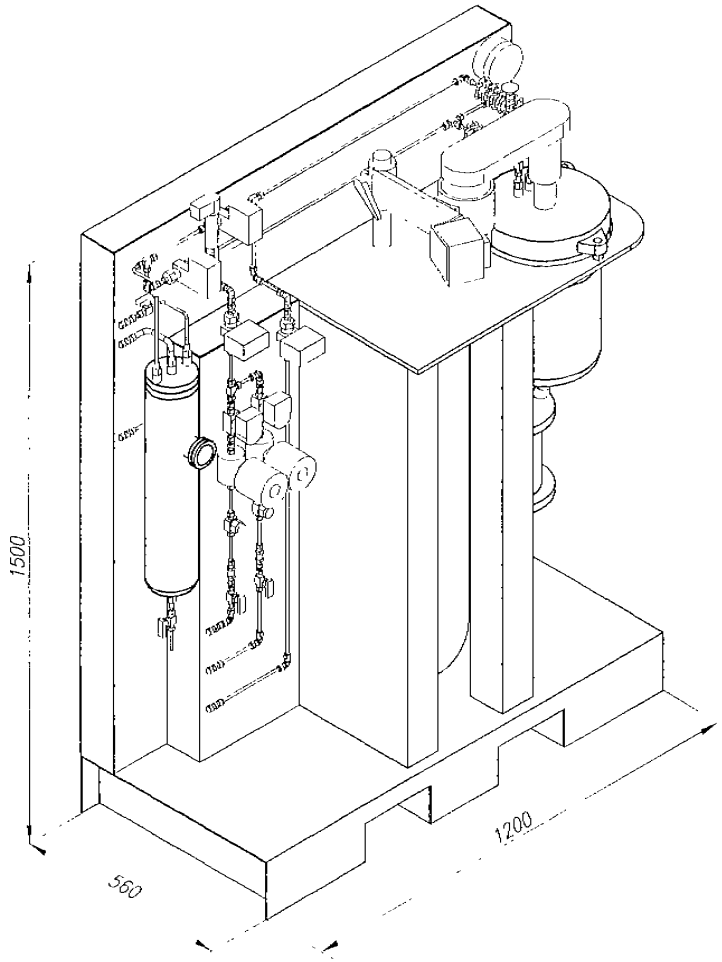

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BIAZZI KILO-LAB UNIT (10L).

Schematic Diagram