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**INTEGRATION AND OPERATIONAL STRATEGY OF A FLEXIBLE  
AUTOMATED SYSTEM FOR SAMPLE ANALYSIS.**

**Denys AR Dugendre**

A Thesis submitted to Middlesex University in partial fulfilment of the requirements for  
the degree of Master of Philosophy

**March 1999**

The was carried out at Rhône-Poulenc Agriculture Ltd, Fyfield Road, Ongar CM5 0HW  
and Middlesex University, School of Engineering Systems, Bounds Green Rd,  
London N11 2NQ

<b>1. INTRODUCTION</b>	<b>8</b>
1.1. RHÔNE-POULENC SA	8
1.1.1. RHÔNE-POULENC AGRICULTURE LTD (RPAL)	8
1.2. THE TEACHING COMPANY SCHEME	10
1.3. PROJECT OBJECTIVES	11
1.4. LABORATORY AUTOMATION AT RPAL	12
1.4.1. COMPANY STRATEGY	12
1.4.2. PROJECT JUSTIFICATION	13
1.4.3. SAMPLE PROCUREMENT	13
1.5. PREVIOUS WORK	14
<b>2. SYSTEM SPECIFICATIONS AND CONSTRAINTS</b>	<b>16</b>
2.1. USER REQUIREMENTS	16
2.2. PROCESS DESCRIPTION & REQUIREMENTS	16
2.3. SYSTEM CONTROL HARDWARE SPECIFICATIONS	18
2.3.1. PROGRAMMABLE LOGIC CONTROLLER	18
2.3.2. ROBOT CONTROLLER	19
2.3.3. COMPUTER NETWORK	19
2.4. REGULATORY REQUIREMENTS	20
<b>3. MODIFIED WORKSTATIONS</b>	<b>22</b>
3.1. WEIGHING WORKSTATION	22
3.1.1. BALANCE	22
3.2. SOLVENT DISPENSER WORKSTATION	23
3.3. LIQUID SCINTILLATION COUNTER WORKSTATION	24
3.4. PIPETTE WORKSTATION	25
3.5. HEATING BLOCK WORKSTATIONS	27
3.6. SOLID PHASE EXTRACTION WORKSTATION	27
3.7. GEL PERMEATION CHROMATOGRAPHY WORKSTATION	28
3.8. CENTRIFUGE WORKSTATION	30
<b>4. PURPOSE BUILT WORKSTATIONS</b>	<b>31</b>
4.1. GENERAL	31
4.2. SOIL EXTRACTION WORKSTATION	35
4.2.1. SOLVENTS	35
4.2.2. MIXING	35
4.2.3. GLASSWARE	36
4.2.4. FILTERING	36
4.2.5. SUB-SYSTEMS INTEGRATION	37
4.2.6. CONTROL/OPERATIONAL STRATEGY	42
4.3. PLANT EXTRACTION WORKSTATION	44
4.4. EVAPORATION WORKSTATION	45
4.4.1. GENERAL OPERATION	46
4.4.2. DETECTION MECHANISM	47
4.4.3. HEATING SYSTEM	49
4.5. VORTEX MIXER WORKSTATION	51

4.5.1.	GENERAL	51
4.5.2.	LABORATORY INSTRUMENTS	51
4.5.3.	MANIFOLD SYSTEM	52
<b>4.6.</b>	<b>ULTRASONIC BATH WORKSTATION</b>	<b>53</b>
4.6.1.	ULTRASONIC BATH SYSTEM	53
4.6.2.	MANIFOLD SYSTEM	53
4.6.3.	FLUIDIC AND AIR SYSTEM	54
<b>4.7.</b>	<b>CENTRIFUGE BALANCING WORKSTATION</b>	<b>54</b>
4.7.1.	GENERAL	54
4.7.2.	MANIFOLD SYSTEM	55
4.7.3.	WATER DISPENSING SYSTEM	55
4.7.4.	WEIGHING SYSTEM	55

---

**5. OVERALL SYSTEM INTEGRATION** **57**

<b>5.1.</b>	<b>GENERAL</b>	<b>57</b>
<b>5.2.</b>	<b>MANIPULATOR AND TRACK</b>	<b>57</b>
5.2.1.	GENERAL	57
5.2.2.	OPERATION	58
<b>5.3.</b>	<b>GRIPPER DESIGN</b>	<b>59</b>
<b>5.4.</b>	<b>GLASSWARE</b>	<b>60</b>
<b>5.5.</b>	<b>SYSTEM POWER SUPPLIES</b>	<b>61</b>

---

**6. IMPROVED OPERATIONAL STRATEGY** **63**

<b>6.1.</b>	<b>MANIPULATOR CONTROL &amp; OPERATION</b>	<b>63</b>
<b>6.2.</b>	<b>PICKING AND PLACING VESSELS</b>	<b>64</b>
<b>6.3.</b>	<b>CELL LAYOUT AND PROCESS OPTIMISATION</b>	<b>66</b>
6.3.1.	PROCESS MODELLING	66
6.3.2.	ROBOT SCHEDULING (TASK PLANNING)	67
6.3.3.	EVALUATION OF LAYOUTS	67
6.3.4.	PARAMETERS TO VARY TO REDUCE CYCLE TIME	68
6.3.5.	RESULTS	68
<b>6.4.</b>	<b>SYSTEM OPERATION</b>	<b>69</b>
6.4.1.	GENERAL	69
6.4.2.	GRAPHICAL USER INTERFACE (GUI)	70
6.4.3.	DATABASE SYSTEM	71
6.4.4.	RUN-TIME PROGRAMS	72
6.4.5.	SYSTEM RESPONSES TO ERRORS	73
<b>6.5.</b>	<b>DECISION TREES - EXPERT SYSTEM</b>	<b>76</b>
<b>6.6.</b>	<b>SYSTEM ACCESS CONTROL AND EMERGENCY STOP</b>	<b>78</b>

---

**7. DISCUSSION** **81**

<b>7.1.</b>	<b>OPERATOR VS DESIGNER RESPONSIBILITIES</b>	<b>81</b>
<b>7.2.</b>	<b>THROUGHPUT AND PERFORMANCE</b>	<b>82</b>
<b>7.3.</b>	<b>PROJECT IMPACT AND KEY LEARNING</b>	<b>84</b>
7.3.1.	AT COMPANY LEVEL	84
7.3.2.	AT PERSONAL LEVEL	84

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**8. CONCLUSIONS** **85**

**9. REFERENCES** **89**

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**10. APPENDICES** **92**

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<b>10.1.</b>	<b>APPENDIX A: PUBLISHED WORK</b>	<b>92</b>
<b>10.2.</b>	<b>APPENDIX B: MECHANICAL DRAWINGS</b>	<b>112</b>
<b>10.3.</b>	<b>APPENDIX C: ELECTRICAL &amp; PNEUMATIC DRAWINGS</b>	<b>151</b>

## **Abstract**

This project describes the integration of a twenty-two workstation laboratory automation system based around a track-mounted robot. The required level of operational flexibility of the overall system puts the emphasis on interfacing and controlling effectively a large number of instruments. The integration of such a large system can sometimes be tricky as off-the-shelf instruments are put near side modified commercial equipment and purpose built workstations.

The automated system being developed at Rhône-Poulenc Agriculture Ltd (RPAL) automates several highly manual analytical processes following one another in a constantly varying order. The integration of such a system was carried out in collaboration with a software and a mechanical engineer. The choice of the control system was made so that the variety of workstations included could be controlled by a limited number of different means. After having drawn the specifications and estimated the number of inputs/outputs needed for every station, a PLC was acquired together with four computers. Various electronics interfaces had to be built or purchased in order to fully operate the system from the controlling computers. Printed circuit boards have been designed and manufactured at Middlesex University together with many mechanical parts for different stations. This integration had to make sure that the system will operate as intended and governed by the parameters entered by the user. System's behaviour and safety in case of an error or an emergency was studied and an emergency stop circuit together with interlocks was implemented. The PLC program was designed so that the machines will fail safe in case of a problem. Being a tool for method development and optimisation, the system evolves gradually towards becoming an expert system. From the information gathered during runs, a decision tree is implemented and responsibilities are gradually withdrawn from the user.

Cross-contamination, radio-labelled samples, and solvent compatibility are determining factors in the safety evaluation and validation processes.

This system was developed as part of a three year Teaching Company Scheme collaboration project between Middlesex University and RPAL. The diversity of the task required the participation of three engineers with varying skills: mechanical, software, and electronics.

## List of Figures

Figure 1: TCS Associates Responsibilities	11
Figure 2: Sample Procurement	13
Figure 3: Information Triangle	17
Figure 4: Control System Distribution	17
Figure 5: Automated System Control Cycle	18
Figure 6: Workstation Control Hierarchy	18
Figure 7: Automated System Control Layout	19
Figure 8: Solvent Dispenser	23
Figure 9: Last Drop Collection System	24
Figure 10: SDW Controlling Signals	24
Figure 11: LSC Improved Duplex Communication	25
Figure 12: Pipette Workstation Solvent Paths	26
Figure 13: PW & RC Improved Communication	27
Figure 14: SPEW Improved Communication	28
Figure 15: GPCW Control Signals	29
Figure 16: GPCW Improved Communication	29
Figure 17: Control Structure	32
Figure 18: Station Status Modes Switching Process	33
Figure 19: Soil Extraction Workstation	35
Figure 20: Glassware used in the SEW	36
Figure 21: Limit sensors used in the Soil Extraction Workstation	39
Figure 22: Soil Extraction Workstation Connections	42
Figure 23: Filtration Process Simplified Algorithm	43
Figure 24: Pressure variation during filtration	43
Figure 25: Plant Extraction Workstation	45
Figure 26: Plant Extraction Workstation Connections	45
Figure 27: Evaporation Workstation	46
Figure 28: Evaporation End Point Detection Principle	47
Figure 29: End Point Detection Electronics Interface	48
Figure 30: Vortex Mixer Workstation	52
Figure 31: Inductive Sensor used in VMW	53
Figure 32: Ultrasonic Bath Workstation Fluidic and Air System Connections	54
Figure 33: Centrifuge Balancing Workstation Layout	56
Figure 34: Design Cycle	57
Figure 35: Kinematics of the CRS manipulator	58
Figure 36: Robot Gripper	59
Figure 37: Glassware	61
Figure 38: Safe positions on robot track	64
Figure 39: Pick-and-place sequence for normal racks	65
Figure 40: Pick sequence for terraced racks	65
Figure 41: Place sequence for terraced racks	65
Figure 42: Optimised Cell Layout	69
Figure 43: Method Designed using the GUI	70
Figure 44: Database Structure	71
Figure 45: Interaction of run-time programs and instrument software drivers	72
Figure 46: System Error Behaviour	76
Figure 47: Sample Extraction Strategy	78
Figure 48: Access Control Process Flow Chart	79
Figure 49: The basic elements of supervisory control (after Moray, 1986)	81

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My thanks and appreciation also go to Dr Raj Gill, Dr Mehmet Karamanoglu and especially Dr Jeremy Lewis who participated on a day-to-day basis in the project design and implementation without whom this project would not have succeeded.

Finally, many thanks to Mr Brian Nuttall of the Teaching Company Directorate for his support and advice.



## 1. INTRODUCTION

### 1.1. Rhône-Poulenc SA

The company that was to become Rhône-Poulenc began life in the 1830's amidst the humble surroundings of a silk-dyeing workshop in Lyon, France. The company's involvement in plant protection began several years later with the manufacture of products to control powdery mildew on grape vines, and after several mergers, the company became known as S.C.U.R (Société Chimique des Usines du Rhône).

Meanwhile, in Paris, pharmacist Etienne Poulenc and his brothers formed 'Les Etablissements Poulenc Frères' in 1900, and in the early 1900's French and British chemical activities came together when the Poulenc-Frères sub-contracted May & Baker to supply carbonate and other lithium salts.

Over time, S.C.U.R. merged with several other companies, including in the late 1920's the Poulenc-Frères, and eventually, in 1961, the mergers produced the holding company known as Rhône-Poulenc S.A.

Active in 160 countries, Rhône-Poulenc is now a global company which ranks in the top seven pharmaceutical and chemical Groups worldwide, with leading positions in each of its core businesses. Worldwide sales top £10 billion.

*"The Group's strategy, at the dawn of the third millennium, focuses on achieving growth through innovation and globalisation, with particular emphasis on creating value, professionalism and customer service." Jean-Rene Fourtou, Managing Director.*

#### 1.1.1. Rhône-Poulenc Agriculture Ltd (RPAL)

The crop protection business of Rhône-Poulenc Agriculture Ltd in the UK draws upon and contributes to the strength of the Rhône-Poulenc Group, providing solutions to farmers the world over. The Company is able to use its wealth of experience to support the continuous programme of innovation that has established its enviable reputation.

A world leader in plant health, Rhône-Poulenc Agriculture Ltd not only researches and develops successful new products, but is also aware of its continued responsibilities to the environment and the maintenance of wildlife populations.

All compounds that pass through the Company's laboratories undergo extensive research to make sure that they are safe in every possible way. It is the Company's duty to the environment to make sure that adverse effects are minimised and to avoid upsetting the balance of nature. Environmental studies are carried out on products to trace what happens in soil, water, flora and fauna.

In the UK, RP Group employs over 4000 people who work at more than 20 locations on products for both home and world markets. We manufacture at 13 of these sites and also have two world class Research & Development facilities at the leading edge of pharmaceutical and agricultural research. The UK research centre based in Ongar, Essex, is key to Rhône-Poulenc's continued record of innovation.

In the world of agriculture, many crops are able to benefit from the protection of herbicides, fungicides and insecticides. Rhône-Poulenc Agriculture Ltd produces a wide range of crop protection products, carefully developed to meet the exacting needs of the farmer. Plants can become damaged, weak or stifled through a whole host of external forces whether it be attack by pests, fungal infection or being in competition for nutrients in the soil from weeds. By developing these products, Rhône-Poulenc Agriculture Ltd can help farmers to grow healthy crops and protect them from pest or fungal damage.

Many of Rhône-Poulenc Agriculture's products are brand leaders such as TEMIK®, Diflufenican®, and ROVRAL®, helping to make the Company a leading force in world and European markets. All of the Company's products undergo many years of experimental tests prior to release into the public domain. The essential research and development and active ingredient manufacture is co-ordinated between three main centres in the UK, USA and France.

Crop protection has developed over the years into an exacting science. Experience has enabled application techniques to be finely tuned so that minimal amounts of product be

applied at exactly the correct dosage rates. Developments have also made it possible to build plant resistance, so that they can be treated with products that previously would have killed them.

Rhône-Poulenc Agriculture Ltd's business is the care, protection and improvement of plants, enabling them to be healthier, stronger and produce greater yields. The world's population is increasing at a rapid rate and the company believes that chemical solutions to biological problems will help to meet the growing demands placed on food producers across the globe. Although the business plays a very significant part in improving crop yields, the company does not dismiss other approaches to growth and actively encourages the development of best practices in traditional farming methods.

The vision of RP Agro is to be one of the major world leaders in crop protection by being the supplier of the most innovative solutions:

- bringing value to the farmers
- increasing the quality and quantity of crop commodities
- respecting people and the environment

whilst conducting business within the Rhône-Poulenc Group's management principles and values.

## ***1.2. The Teaching Company Scheme***

The mission of the Teaching Company Scheme (TCS) is to strengthen the competitiveness and wealth creation of the UK by the stimulation of innovation in industry through partnerships between academia and business. TCS is supported and financed by a number of government agencies, known as sponsors. For the work described in this thesis, the programme was jointly funded by the DTI and RPAL.

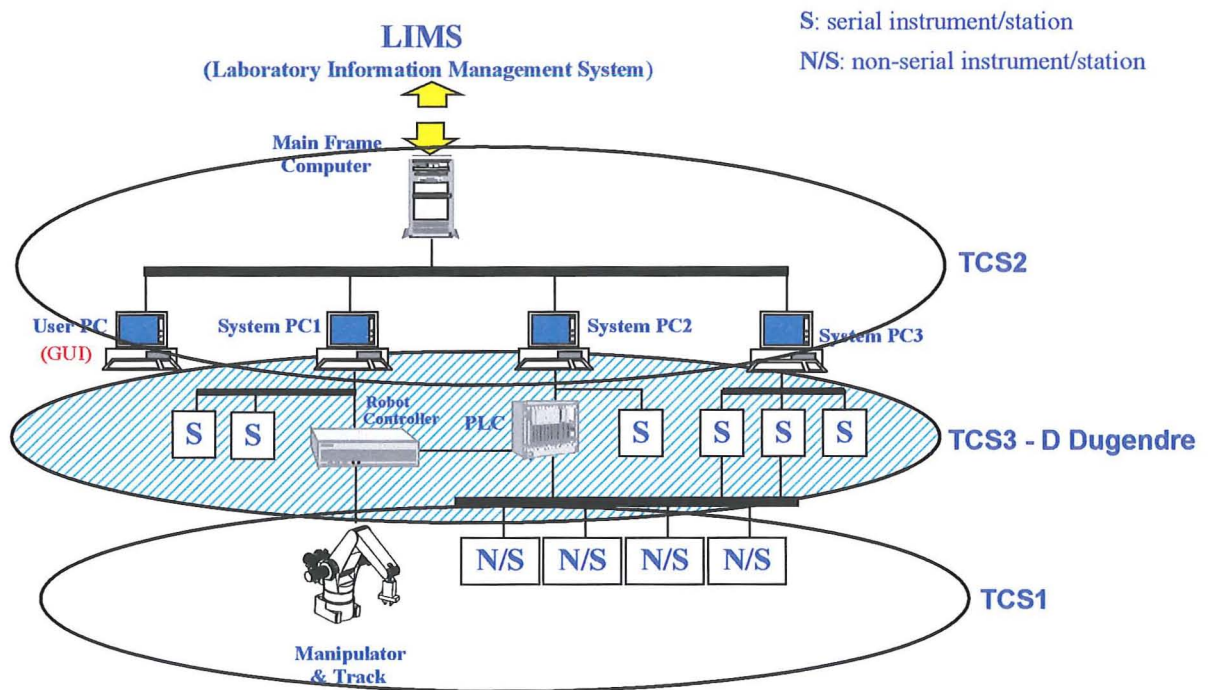
The Teaching Company Directorate (TCD) comprises a number of regional Teaching Company Consultants who are responsible for giving general support and advice to the programme participants. The programme participants are: The Teaching Company Associate (TCA), The Company and the University. The Teaching Company Consultant was Mr Brain Nuttall.

The emphasis on industry was appealing to RPAL with the anticipation of the automation knowledge they lacked and needed.

Each programme has a written proposal called the Teaching Company Programme (TCP) covering the details of the work to be carried out, the personnel involved and the time scales for the completion of each stage for each Associate.

### 1.3. Project Objectives

This TCP involved three Associates over 3 years. Two Associates in the first year, three in the second year and one in the last year.



**Figure 1: TCS Associates Responsibilities**

The general project objectives were to design, build and commission all software and hardware for an automated system for soil or plant sample analysis.

The specific objectives for me for this project were to integrate the supervisory software program and Graphical User Interface (GUI) developed mainly by Teaching Company Scheme Associate (TCSA) 2 and the mechanical workstations developed mainly by TCSA 1 in order to make the system work. An operational strategy was also put in place to make sure that the system would operate safely and following pre-defined conditions. This was achieved by:

- undertaking the design, development and construction of all mechanical and electrical interfaces for a robot system
- undertaking the design, development and integration of all electrical and electronic components of the robot system
- devising and implementing an operational strategy for safe operation of the system

Some understanding of other Associates' work was also a prime objective as my task was to translate the parameters entered by the user through the front end into meaningful signals using chosen sensors and actuators to control the mechanical process. A significant overlap between my knowledge and other Associates' knowledge meant that I was able to carry out software or mechanical tasks to bring the whole system together.

The main objectives listed above were broken down into smaller tasks:

- Conduct review of system specifications and safety requirements
- Assist TCSA 1 and 2 with system build
- Produce system operational strategy and hierarchy of control
- Draw up system controller specifications for robot and workstations
- Design, manufacture and source electronic interfaces for all the workstations, robot and controller
- Write software for workstation control
- Carry out full system test, commissioning and training of the operators
- Write technical manuals (Instruction, Service, Trouble-shooting and Hardware details manuals)

## **1.4. Laboratory Automation at RPAL**

### 1.4.1. Company Strategy

Laboratory automation has shown a wide variation in the success of various systems. Laboratory automation cannot be considered a mature technology in the same way that flexible automation is now accepted in the manufacturing industry. There is almost certainly a greater risk in not going ahead with automation projects, as competitors are carrying out laboratory automation work and gaining experience (Smartt, 1995). The technology involved is maturing rapidly, and to stay competitive in chemical research it is vital for a company not to get left behind. For this reason, RPAL has decided to invest heavily in laboratory automation systems. Gradually, automation has moved from being

a support science, here to sustain the business, to being an integral part of the company's core business.

### 1.4.2. Project Justification

Rhône-Poulenc's Environmental Chemistry Laboratory requires an automated system for sample analysis to improve their method development capability. The company have tried to acquire the technology described in this thesis, but commercial system builders lack the know how, correct mix of skills and the systems integration expertise required for this project. The automated system is required for method development, validation and routine analysis. This will free highly skilled laboratory personnel from conducting tedious and repetitive tasks, and thus make them available for more challenging work. The company's products need to be registered before they can be manufactured and marketed. The registration process requires the environmental fate of the active compounds to be studied extensively. Products have a limited life and need to be replaced continuously with better products. The lead time for a new product can be seven years from discovery to launch. The proposed robotics system will speed up the method development capability for reduced lead times whilst simultaneously improving quality.

### 1.4.3. Sample Procurement

Figure 2 describes the various sources of soil or plant samples which enter the Environmental Chemistry Department at Ongar and are therefore potential candidates for analysis on the automated system.

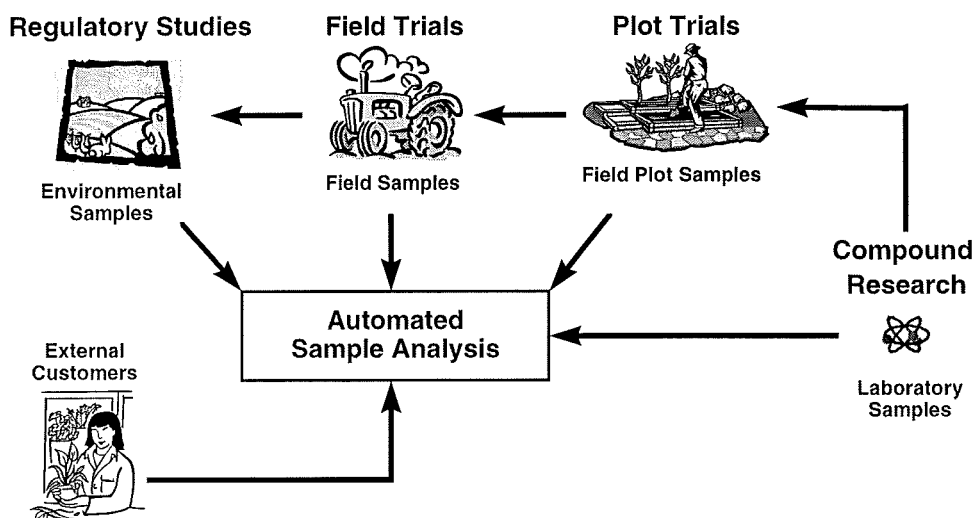


Figure 2: Sample Procurement

The discovery process always starts with a new active ingredient which has been screened amongst millions and has proven to be effective at the right dose. During this screening period, some soil and plant samples will be generated and analysed. Once the candidate compound has successfully pass this stage, it will be tested in the real world during "plot trials". Over 10,000 plot trials are carried out every year. The next step is to test this very promising compound on a larger scale and under various weather conditions. "Field trials" are carried out in special test farms around the world to test for environmental behaviour and degradation under different conditions.

The final step for our candidate compound is to have its environmental fate "profile" approved and registered for trading in a given country. Regulatory studies vary from country to country and the compound "profile" built up during development is not always sufficient to be registered and some additional studies have to be carried out at this stage.

The last source of samples for our system is the studies RPAL is contracted to carry out for external customers.

It has been envisaged that, out of all the samples entering the Environmental Sciences Department at Ongar, up to 70% of them could be processed by the automated system.

### **1.5. Previous Work**

Automation often requires the changing of traditional manual procedures so that they can be automated more easily. Method development is usually done off-line using traditional manual procedures, and then transferred to the robotic system, which can result in problems. As a consequence, the time taken to establish a new automated method is often considerable. Re-validation is then required, in the automated system, before routine analysis can commence. In addition, the sample preparation stage of an analytical method has always been a limiting step in automation, due to the difficulties associated with its automation. Although this step has been automated, it was with limited control of the process (Laws, 1987 and 1988). Thus the need to develop analytical methods, on robotic systems, is vital, especially if flexibility is required. These problems, and numerous others, have limited the effectiveness, so far, of automation in analytical chemistry applications.

The use of robotic systems in laboratory applications has been reviewed by Majors and Crook in 1993. There are numerous references to the use of Zymark robotic equipment

for the analysis of pesticide residues between 1985 and 1997, including Laws and Jones in 1988, and Owens in 1989.

The advancement of computers, robotics and control systems has, in recent years, allowed rapid advances in laboratory automation. Stand-alone automated pipette and solid-phase workstations, as well as analytical quantification techniques (Majors, 1993), are available commercially from several manufacturers and have, over the last decade, made significant impacts into the analytical laboratory. However, stand-alone workstations invariably have not been designed to be integrated into larger robotic systems, and can be deficient in several areas. Most notably communications, robot access, and safety control are problem areas, requiring specialist engineering knowledge. To achieve satisfactory reliability for a complex system, the reliability over normal use of individual components or workstations need to be improved in order to minimise accumulative errors reducing the overall reliability of the system. At minimum, they may require some modifications by the equipment manufacturer, and further in-house customisation, to enable some form of safety control in the automated system. The available access area for samples may not be suitable for the robot, and some further customisation may be required to the equipment, or robot gripper, in order to access the sample area.

Most of the systems developed for the industry are based on fixed or sequential processes (Sommer, 1998) where re-programming flexibility is not necessary. As the system developed at RPAL was going to be used as a method development tool, the possibility of changing parameters and process sequences between two consecutive samples without having to re-program the system, was a determining option in the project implementation. The uniqueness of this system also comes from the fact that each step of the analytical process can take a variable amount of time to be completed and several samples can be analysed at the same time following different processes. The amount of workstations to integrate around the 6-metre track mounted robot in order to carry out the required processes has never been done before in this industry. Huge installations using similar equipment have been implemented at North West Water (Cockburn-Price, 1995), where 11 robotic cells are used to analyse water samples. However, the amount of workstations in any given cell did not exceed the 21 proposed for the automated analysis system at RPAL.



## **2. SYSTEM SPECIFICATIONS AND CONSTRAINTS**

### ***2.1. User Requirements***

The main requirement as far as the user goes is the level of flexibility and friendliness the system must have in order to be easily operated and understood by the user (Cockburn-Price, 1995). This leads to the idea of having a Graphical User Interface (GUI) as a front end to the system. This interface must allow the user to run the system with minimum training and supervision. The GUI must be a good representation of the desired process (Diaz-Cachero, 1997).

The constraints put on the system by the user are sometimes referred to as operational parameters. The operator is currently prompted to enter the method the sample has to follow and the required parameters for each workstation used in the method. The workstations and their parameters must reflect the user needs. Parts of the manual process can actually be improved and/or changed to better fit automation. This is not an easy task and scientists are often reluctant to change the way they work and a fear factor can easily be detected when it comes to experiment with an automated yet complex piece of equipment. Culture was therefore one of the first tasks we had to tackle in order to effectively specify, and later use intuitively, the automated system. Often, compromises had to be made and views had to be challenged, in order to identify the real needs behind the image of the process given by the scientists.

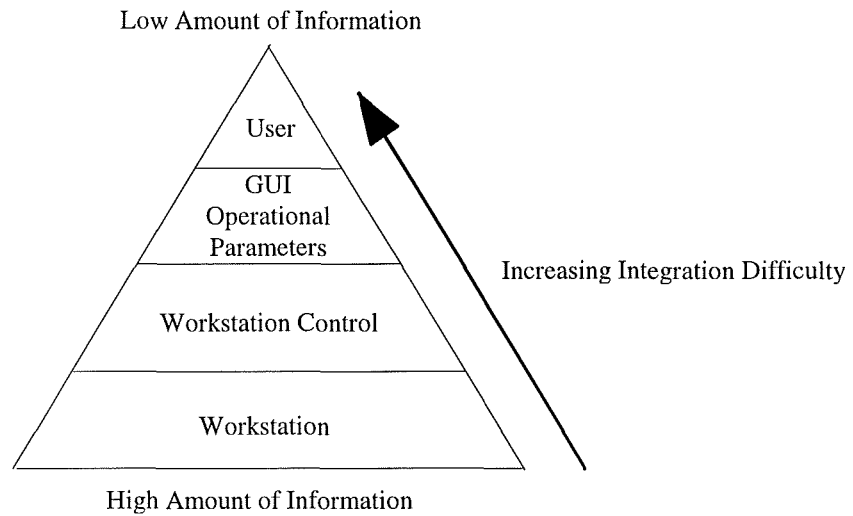
### ***2.2. Process Description & Requirements***

The system integration brings together the user requirements and the hardware capabilities. This is an extremely important step in the design and development stage of the system.

During the integration process the following points were addressed with the highest priority:

- the operational parameters required by the user can be achieved with the hardware selected and with a given accuracy.
- the hardware selected gives some feedback to the user or the supervisory control system.
- the physical interactions between the robot and the various hardware are possible.

### Amount of Information Required for Operation



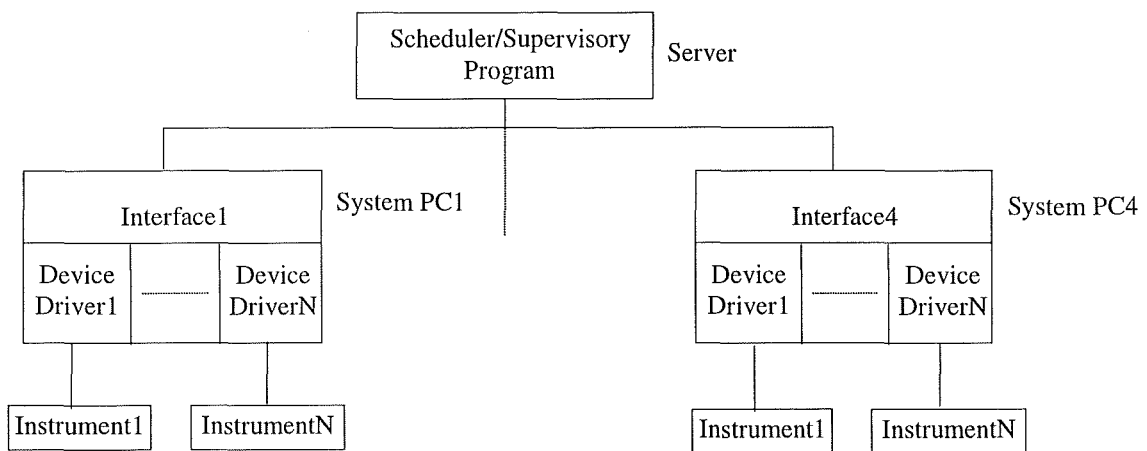
**Figure 3: Information Triangle**

As we get closer to the base of the triangle (see Figure 3), the level of information increases, while its complexity decreases:

- User: Extract this sample with Acetonitrile and mix for 20 mins at high speed.
- GUI Operational Parameters: Solvent density, select solvent pump no2, pumping time, select stirrer speed 8, start signal.
- Workstation Control: All routines and sub-routines to start, operate and stop the station.
- Workstation: Start/stop pump, on/off relay, increase/decrease voltage.

The ultimate aim for the system is to operate autonomously with minimum intervention from the user (reduce user constraints to a minimum), Crook, 1992 and 1993.

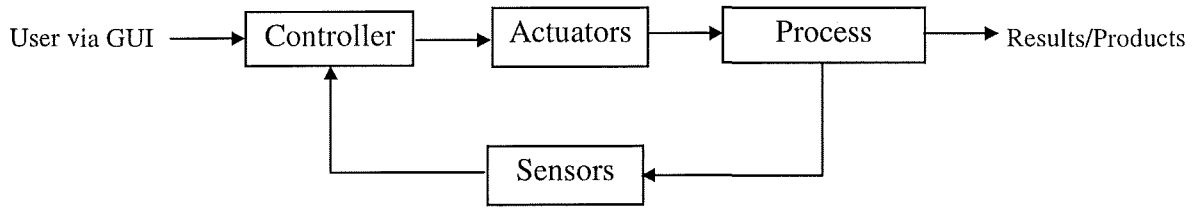
My task was to concentrate on the bottom two items at the base of this information triangle, making sure it will integrate with the GUI and supervisory program by writing some of the device/instrument drivers as shown on Figure 4.



**Figure 4: Control System Distribution**

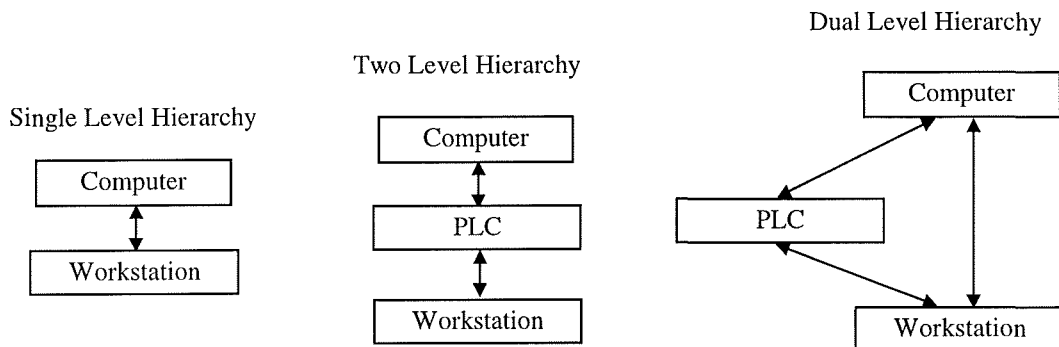
### 2.3. System Control Hardware Specifications

The control hardware consists of a network of five computers, a Programmable Logic Controller (PLC) and the Robot Controller. These are used to control all instruments, workstations and robotic arm.



**Figure 5: Automated System Control Cycle**

The automated system control cycle (Figure 5) describes the approach taken in most cases. The ‘Controller’ stated on Figure 5 acts as a logic comparator which will power actuators to influence the process according to the state of the sensors monitoring the process. This ‘Controller’ role is played by either the PLC, a computer or both (see Figure 6) depending on the type of station on which the process is taking place.



**Figure 6: Workstation Control Hierarchy**

Dual level hierarchy is the most difficult one to synchronise as it requires both PLC and Computer for the workstation to work. Out of all the off-the-shelf equipment brought in, only two were fully controllable by a computer with no customisation at all. Most of the other instruments had to be modified slightly so that the level of control or feedback could be improved by using the PLC.

#### 2.3.1. Programmable Logic Controller

The PLC, programmed in Ladder Logic, is used to control custom designed and built workstations, and to interface them to the computer network. The PLC used is an Omron Sysmac C200H with digital and analogue I/O modules.

This PLC consists of a rack mounted CPU with an 8-slot back plate, two expansion racks featuring 8 slots as well, which represent a total of 24 individual I/O modules. They include: ADC modules, DAC modules, O/P modules and I/P modules.

### 2.3.2. Robot Controller

The pick and place robot routines were written in RAPL-II and stored in the CRS robot controller (model: C500). Teaching of the locations was made easy by the use of the teach pendant.

### 2.3.3. Computer Network

The Network is a thin Ethernet network linking five computers: a “server” (P166), a “user PC” (486), three “system PCs” (486s) running under Windows 3.11 for Workgroups. Drivers for serial equipment and overall control routines were implemented using Borland Delphi as the programming environment and Borland Paradox for the databases. The software has evolved around the concepts of safety, flexibility of operation, and modularity for expansion.

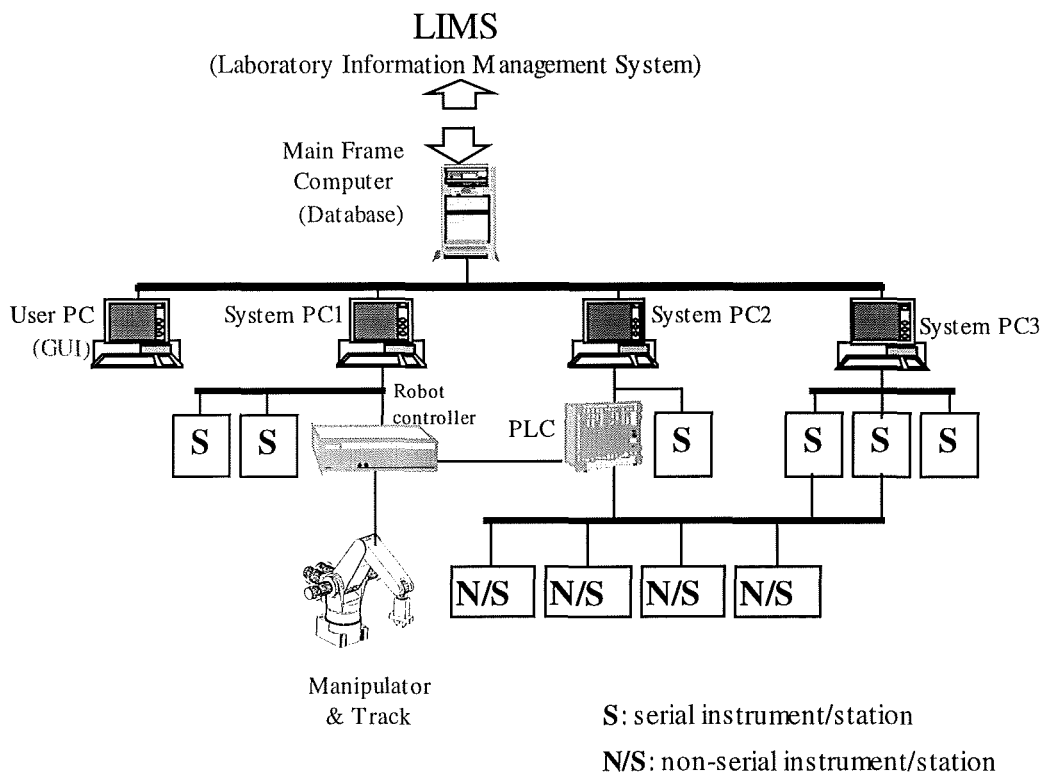


Figure 7: Automated System Control Layout

## **2.4. Regulatory Requirements**

The system must fail safe in case of an emergency or power failure.

The constructed system has to be safe to operate in a laboratory environment. Therefore the design and operation of the system must comply with the regulations and standards listed below (Isenhour, 1989, Jones, 1988 and Schoeny, 1991):

- Control of Substances Hazardous to Health (COSHH)
- Good Laboratory Practices (GLP)
- Fire and Safety Regulations.
- British Standards/European Norms (BS/EN) on safety of machinery and electrical safety.

Shortly after the US FDA (the United States government's Food and Drug Administration) introduced GLP (Good Laboratory Practice) regulations in 1976, the Organisation for Economic Co-operation and Development (OECD) published a compilation of Good Laboratory Practices. OECD member countries have since incorporated GLP in their own legislation.

In Europe, the Commission of the European Economic Community has made efforts to harmonise the European laws. Directives were produced to place obligations on member states to comply accordingly.

Guidelines on quality assurance for measuring equipment and for calibration laboratories have also been published by the International Organisation for Standardisation (ISO) and others.

Other regulations regarding safety of machinery, reliability of systems, equipment and components, industrial robots, safety requirements for electrical equipment for measurement, control and laboratory use must be taken into account when developing a system for automated analysis.

These regulations are published by the British Standard Institute (BSI) in the UK and are referred to as British Standards (BS) or European Standards (EN).

These standards are set by the members of the European Committee for Standardisation (CEN).

In the particular case of Automated Analysis, some of the British Standards listed below should be seen as some kind of framework for implementation.

**BS 5304 : 1988**                      Safety of machinery

<b>BS 5760 : 1993</b>	Reliability of systems, equipment and components
<b>BS 7687 : 1993</b>	Safety requirements for electrical equipment for measurement, control and laboratory use
<b>BS 5345 : 1989</b>	Selection, installation and maintenance of electrical apparatus for use in potentially explosive atmospheres
<b>BS EN 60079-10:1996</b>	Electrical apparatus for explosive gas atmospheres
<b>BS 7228 : 1992</b>	Industrial robots

The most important development in the field of Health and Safety has been the increasing concern on the part of the public in respect of the manufacture, transport and use of chemicals. The latter concerns have led to the Control of Pollution Act in 1990.

The principal legal instrument concerned with Health and Safety at the work place is the Health and Safety at Work Act 1974.

The Act applies to all persons at work: employers, the self-employed, and employees, the only exception being domestic servants in private employment.

The Control Of Substances Hazardous to Health (COSHH) regulations contain an absolute requirement that an assessment of risks to health must be made before any work activity is undertaken using a hazardous substance (Gentsch, 1993).

COSHH assessments should be regarded as establishing good working practices.

### **3. MODIFIED WORKSTATIONS**

The twenty two workstations featured in the RPAL automated system include some off-the-shelf, modified off-the-shelf and purpose built workstations. This section describes the first two categories while the purpose built workstations are described in the next chapter. Out of the total number of instruments used in this system, only two could be readily integrated into the system. All other commercially available equipment had to be modified to a certain extent in order to be fully integrated with the rest. This chapter describes the characteristics of the chosen equipment and the level of customization required.

#### **3.1. Weighing Workstation**

The analytical balance used here has an open weighing pan so that the robot can load/unload it. The dimensions of the pan are sufficient to allow a rack to be fitted to hold the vessels. This is determined by the number, the size and the shape of the vessels to be weighed. The rack currently holds 7 different types of vessels and the calibration weights during the weighing process. The balance is the most often used station of all as weight is measured before and after the sample is processed by any of the workstations. The only modification that was made to this instrument is the holding rack for the various types of vessel which was designed to fit the limited space on the balance pan. Another rack was built to accommodate the calibration weights provided with the balance. The robot is required to stay near the balance while the weight is being measured as it is a swift process.

##### **3.1.1. Balance**

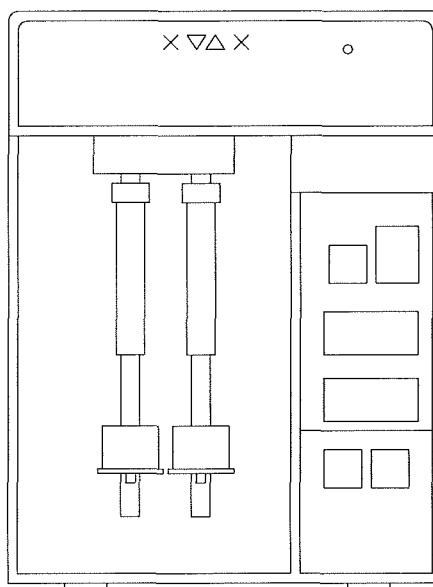
The balance used in this station is a Mettler Toledo PB602. The reason for choosing this balance is its readability to capacity ratio. A readability of 0.01 g is available with this balance. The need for a high capacity and high readability made this balance a suitable candidate. The smallest vial used weighs 2 g. The largest vessel including its contents do not exceed 450 g. The maximum capacity must also include the weight of the rack to be fitted on the weighing pan of the balance (estimated to be around 50 g using nylon). This brings us to a maximum capacity of 500 g.

The balance also features an RS232 interface and is therefore connected directly to a host computer.

### 3.2. Solvent Dispenser Workstation

The Solvent Dispenser Workstation (SDW) is based around a Compudil D from HTI (Hook and Tucker Instruments Ltd ) liquid handler. This liquid handler features a 25ml syringe pump that allows solvent addition to an accuracy of  $\pm 1\mu\text{l}$  at best. The maximum volume to dispense will depend upon the speed of the syringe drive - speed can vary from 2 to 12 seconds for full stroke depending on the level of accuracy required.

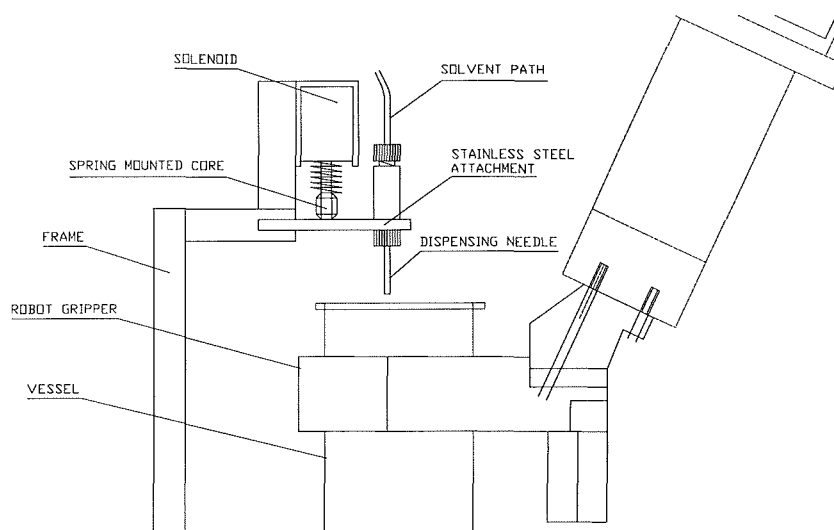
This instrument is fitted with a valve switching unit to allow the user to use different solvents (up to 4). The SDW is the only station with the Weighing Workstation that require the robot to be present during operation. The robot holds the vessel while the required amount of solvent is being dispensed. The time taken by this process does not impact significantly on the overall performance of the system.



**Figure 8: Solvent Dispenser**

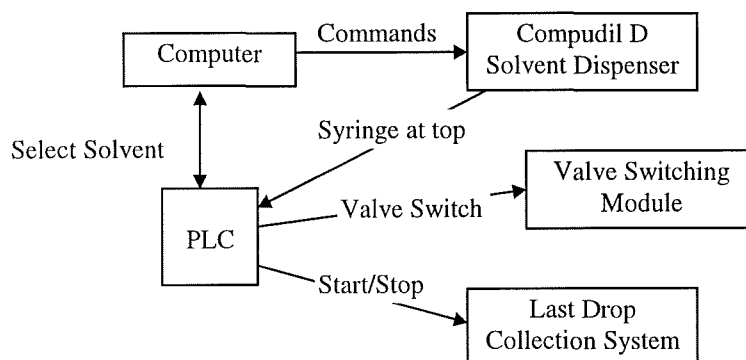
The only modification made to this instrument is the last drop collection system. This system consists of a micro-switch mounted on the syringe drive that detects the end of the stroke and activates a solenoid mounted on the dispensing needle. The solenoid is latched for a few seconds while the needle finishes dispensing. The solenoid is then released and its core is allowed to hit the flexible stainless steel attachment on which the needle is mounted, allowing to collect the very last drop of solvent. See figure below.





**Figure 9: Last Drop Collection System**

The Compudil features an RS232 interface and is therefore connected directly to a computer, but the workstation is also connected to the PLC for the last drop collection system and the solvent valve switching control module.



**Figure 10: SDW Controlling Signals**

### **3.3. Liquid Scintillation Counter Workstation**

The Liquid Scintillation Counter (LSC) is part of the analytical process that will reveal if the active ingredients have been recovered and in what proportion (Donzel, 1993).

The instrument used is a Packard Tri-Carb 1000 Liquid Scintillation Analyser. The LSC provides an indication of the sample radioactivity in compensated Disintegrations Per Minute (DPM) instead of uncorrected Counts Per Minute (CPM) raw data. It also gives an indication of whether the results are homogenous or heterogeneous based on sample quality.

This instrument is a single shot LSC and uses 28 mm vials with screw caps. A 172 location rack is combined with the LSC. Manually, the instrument is loaded at the top,

one vial at the time. On pressing the 'Count' key on the keypad, the vial is lowered inside the LSC and analysed following a pre-entered protocol. The LSC comes with a unidirectional RS232 interface which is fine for reading data from the instrument but does not allow to send commands to it. Only a small number of commands are necessary to operate the instrument. Therefore a few modifications have been carried out on the LSC in order to improve its integration and operation within the automated cell.

1<sup>st</sup> modification: In order to start the LSC remotely, a contact closure output from the PLC was connected in parallel with the 'Count' key on the instrument front panel. Hence the process can be started remotely using the PLC.

2<sup>nd</sup> modification: The data generated during the analysis of a given sample vial is sent to the host computer at the end of the process but before the vial re-emerges from the instrument. This cannot be used as a trigger for sending the robot to unload the vial as the lift mechanism may jam when raising the vial. Therefore a supplemental feedback mechanism was needed to inform the overall process controlling computer that the vial had effectively been raised. Once again this is achieved by mounting a proximity sensor on the lift mechanism inside the LSC casing. See Figure 11.

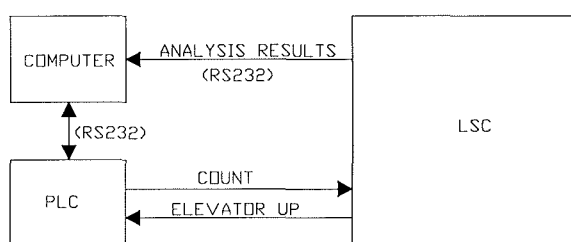
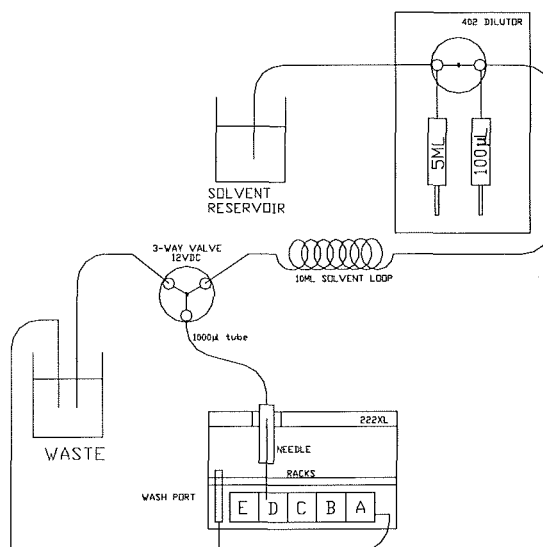


Figure 11: LSC Improved Duplex Communication

### 3.4. Pipette Workstation

The Pipette Workstation (PW) consists of a Gilson XYZ liquid handler (model: 222XL) coupled to a syringe pump module (model:402). The syringe pumps available are: 5ml and 100µl. See Figure 12. This station is used to take sample aliquots for the LSC and transfer given volumes from one test tube size to another.



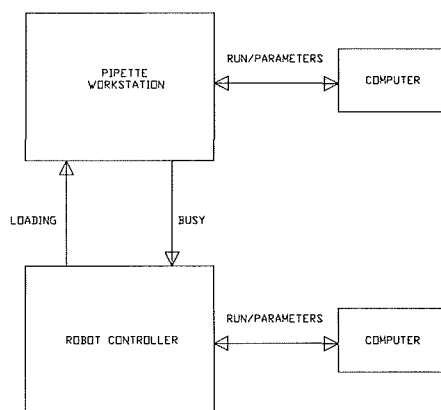
**Figure 12: Pipette Workstation Solvent Paths**

The Gilson 222 XL is controlled directly by a computer via an RS232 bi-directional serial link. This 222 XL central unit controls the 402 Syringe Pump through the GSIOC. A number of valves can be controlled using the open collector output. In our case, only one valve is used. It is connected to pin 5 & 6 of the open collector output.

The process on the PW is as follows: the robot loads the source vessel (from which aliquots will be taken) and the required number of destination vessels or vials. The pipette then operates once the robot is out of the way. In order to make sure that the robot will not unload the vials while the pipette is working, extra handshake signals were required. These signals were implemented at a low level and programmed into the device drivers.

When the robot is required to move, the master computer runs a program inside the robot controller with the required parameters. Similarly, the Gilson device driver provided with the instrument is called up by the main program and run with specific parameters. The extra feedback signals have been implemented so that the robot controller and the pipette driver can take decisions locally without having to refer to the overall control program.

For example, when the robot loads the PW, an output from the robot controller is set to 1 which prevents the pipette from starting. If the pipette is currently running, a relay output is set to 1 which is picked-up as robot controller GPIO input. This will prevent the robot from attending the pipette. See Figure 13 below.



**Figure 13: PW & RC Improved Communication**

### **3.5. Heating Block Workstations**

The Heating Block Workstations (HBW) are used to concentrate the sample by evaporating solvent, similarly to the evaporation workstation, see section 4.4.

There are two HBWs, they consist of two separate heating modules. The smaller one is used for straight forward evaporation of solvent in different test tube sizes. The larger unit will be used to evaporate under favourable conditions down to a minimum volume. This module will operate only with one test tube size and will use a Nitrogen jet to accelerate the evaporation process. This station is still under development as it was a last minute requirement from the users. This larger module will work similarly to the smaller one but it will be connected to the PLC for the control of the manifold and gas system. A possibility of using a level detection system similar to the one used in the EW (section 4.4.2) is also envisaged.

The small HBW is used for straight forward evaporation in various vessel sizes. This station integrates a Liebig heating block fitted with a West Instrument temperature controller. This allows a dual set point to be set remotely through an RS485 current loop interface. In order to interface this block with the host computer, an RS232/RS485 converter was needed. The converter was purpose made as the RS485 interface is a two-wire link as opposed to the more common 4-wire one.

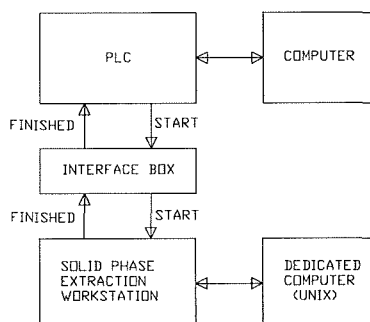
### **3.6. Solid Phase Extraction Workstation**

This workstation is used to separate a solid from a liquid phase in a sample test tube by filtration. The hardware, manufactured by Waters, and software installed on the dedicated computer (UNIX) were already available in the laboratory for integration in the robotics system. The Solid Phase Extraction Workstation (SPEW) consists of two main modules: a transport system and a fluidic module. The transport module is a

compact XYZ module with an integral control panel. A probe mounted on the transport module carries out filtration, solid phase extraction, sample and fluid handling. The probe handles filters and cartridges as well as liquid aspiration and dispensing. All of the necessary solvents, gases, sensor controls and external communication interfaces are provided by the fluidic module.

The fluidics module contains the valves, pumps, power, supply, drivers and controllers used to select and transfer liquids and gases to and from the probe. On the front of this module are the main system power switch, two syringe pumps, a fluid distribution valve, and two solvent selection valves (one for each syringe pump) with LED status indicators.

This station is controlled by a dedicated computer running under UNIX. Protocols for analysis are entered via the station's computer. After careful analysis, it was identified that a unique protocol common to all users can be used as studied by Laws, 1987. The only interaction needed between the SPEW and the overall system control computer is therefore a start and stop signal currently generated by the PLC via a relay interface box. See Figure 14.



**Figure 14: SPEW Improved Communication**

### **3.7. Gel Permeation Chromatography Workstation**

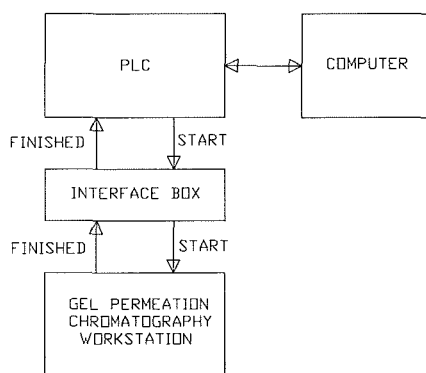
The Gel Permeation Chromatography Workstation (GPCW) allows to separate the individual products in a mixture by passing it through a column where different products will migrate at different speeds. The fractions of each individual products are then collected for further analysis.

This workstation includes the following modules:

- Gilson 401 Dilutor
- Gilson 221 Auto-sampler
- Gilson 202 Fraction Collector
- 6-way universal switching valve

- Gel Permeation Column (Bio-beads SX-3)
- HPLC pump
- Two 3-way solenoid valves

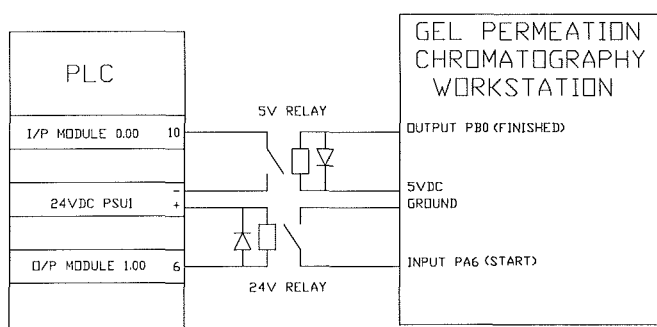
Similarly to the SPE Workstation, the GPC runs a unique protocol suitable for all users as demonstrated by Wright, 1991, Millier, 1992 and Majors, 1993. This protocol is pre-programmed and only a start and finished signal is needed to/from the GPC for remote operation. See Figure 15.



**Figure 15: GPCW Control Signals**

The GPC protocol on the Gilson 221 auto-sampler has been modified so that the robot can be called on completion of the program and reversibly, a start signal can be sent to the GPC once all test tubes have been loaded. The program has also been modified so that it injects from a rack location accessible to the robot. The fraction collector protocol has been changed to collect 10 fractions into standard laboratory glassware, all at rack locations accessible to the robot.

An interface is required between the GPC and the PLC in order to receive and generate the extra start and finished signals. The PLC I/O modules are supplied with 24V, but the GPC can only accept a 5V signal, therefore a very simple voltage change circuit was designed using two relays of different rating. See drawing in appendix.



**Figure 16: GPCW Improved Communication**

### **3.8. Centrifuge Workstation**

The centrifuge is used to separate two layers or phases of different densities in a sample. The centrifuge was required to accept three different types of vessels. Hence specially made solvent resistant inserts were fabricated to fit the rotor buckets.

The chosen centrifuge is a Sigma 6K10 with refrigeration unit and a max. speed of 4500 rev/min. This centrifuge was chosen due to its small bench top size (as opposed to self-standing ones). The need for an entirely automated instrument led us to choose this particular product as the supplier offered us to make the required modifications to the instrument following our requirements: the centrifuge rotor was required to always stop in the same place with a brake applied so that the robot can load/unload the buckets. It was also required that the lid opens/closes automatically on finishing/starting the process. All other parameters can be set and changed via the optional RS232 interface purchased with the instrument.

The centrifuge is controlled directly by a computer which sets the user parameters prior to running it. The lid opening/closing mechanism is controlled by the PLC once a start confirmation signal has been sent by the overall system control program. The rotor indexing mechanism is internal to the centrifuge and is operated automatically when the lid is requested to open. This indexing can be overridden by an external switch on the side of the instrument for stand-alone use.

The interface between the PLC and the centrifuge for the lid open/closed signals is a simple relay box. The micro-switch for lid open and lid closed states are mounted on the linear actuators inside the casing and are connected directly to two PLC inputs. One side of the switch is supplied with 24V while the other side is connected to the ground via a PLC input. The 'open lid' and 'close lid' signals are generated by the PLC as digital 0-24V signals, therefore a relay is needed for interfacing the signals with the instrument keypad. Two 24V SPNO reed relays with reverse current protection are used for this purpose.

## 4. PURPOSE BUILT WORKSTATIONS

This section highlights the relation between the operational strategy and the hardware requirements to achieve correct operation for this type of hardware.

### 4.1. General

The purpose built workstations title refers to stations that have undergone a major development or adaptation in order to become 'robot friendly'.

The mechanical structure of these stations was designed and built in-house in collaboration with a mechanical engineer. A workstation is formed by several sub-systems integrated together. The sub-systems are usually divided into four categories:

- Instrument System: such as the stirrer, macerator, etc.
- Manifold System: used to handle vessels or secure vessels in place
- Fluidic System: solvent paths
- Air System: vacuum and compressed air lines

In order to integrate 22 workstations into a single work cell, some sort of handling mechanism was required.

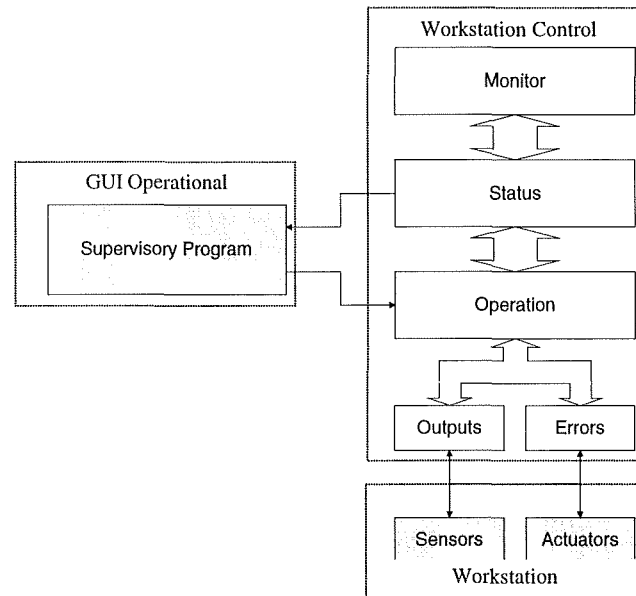
The choice of a suitable robot to make the link between the different modules was tremendously important and certain parameters determined the right one for the job: dexterity, workspace, payload, and most importantly: the gripper which will mechanically interface directly the glassware with the workstation (Laws, 1988).

Further integration came from a limited number of vessels used in the system.

Integration, design and operation are three indistinct notions evolving in parallel. If one is modified, the others will be affected to a certain extent. We should try as much as possible to make the design and integration of a piece of equipment flexible so that its operation can be changed slightly without having to re-design the whole machine. The only problem with this policy is that it takes time, effort and resources to achieve a certain level of flexibility. Therefore, it is sometimes difficult to know where to draw the line. This is one of the major problems I have encountered in this project.

The operational strategy has several layers that correspond to various sub-routines in the station control structure. See Figure 17.





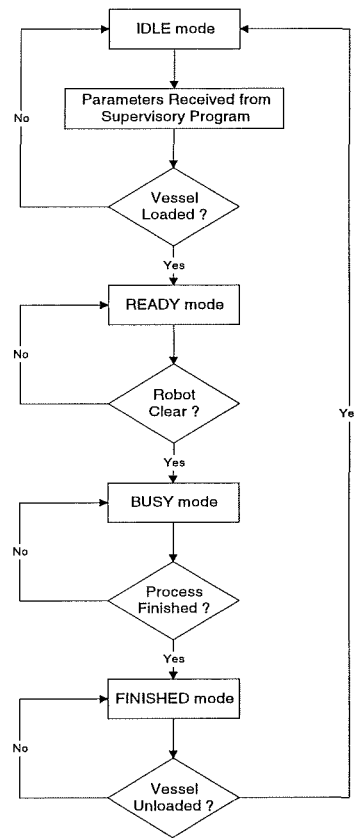
**Figure 17: Control Structure**

For a workstation to start working, it needs three different inputs from other elements of the system:

1. The operating parameters: they are provided by the supervisory program based on the GUI entries. It will not be downloaded to the station control if busy.
2. The confirmation signal: once again, this is provided by the supervisory program as a unique flag to confirm that all parameters have been downloaded successfully and that the robot is going to be sent for loading the vessels.
3. The start signal: it is sent by the robot controller through one of the PLC inputs once the robot has loaded all required vessels and is clear off the station.

Providing that the station has received the above in the right order, then the process will start.

During operation, the workstation will go through various status, errors and process flags. A basic structure of routines is common to all workstations. They include: a *Status* routine for external monitoring by a host computer running a supervisory program, a *Monitoring* routine for internal monitoring, an *Operation* routine for normal operation handling, an *Outputs* routine for handling the actuators, and an *Errors* routine for faults and recovery actions.



**Figure 18: Station Status Modes Switching Process**

**Status:** Five different status are used in most of the workstations. They are: IDLE, READY, BUSY, FINISHED, ERROR. These status words are used for external monitoring (monitoring by the supervisory program), see section monitor below for more information.

If the station is not in use then the status will be “IDLE”. Once it has received the operational parameters and the confirmation signal, its status changes to “READY”. When both vessels have been loaded and the robot controller has sent the start signal, the status switches to “BUSY”. It will stay “BUSY” until the process finishes or an error is detected in which case, the status “ERROR” is flagged. At the end of the process, the station returns the vessels to the loading/unloading position at which stage, the status becomes: “FINISHED”. The station will go back to the “IDLE” status only when all vessels have been unloaded. See Figure 18.

**Monitor:** Monitoring routines are at two different levels: external and internal monitoring.

The external monitoring informs the supervisory program on the current status of the station/process upon which some decisions might be taken. The internal monitoring acts as a base to build a higher hierarchical level especially when it comes to error recovery.

One Word is used for each station for external status monitoring. The different bits inside this word will differentiate between the different status.

Example: Word 30 contains stirrer status bits. During the process, the supervisory program will monitor if the station has finished working by enquiring word 30, if the value is 8 then the stirrer has finished.

Word	Bit	Meaning	Value
30	00	Stirrer IDLE	0
30	01	Stirrer READY	2
30	02	Stirrer BUSY	4
30	03	Stirrer FINISHED	8
30	04	Stirrer ERROR	16

Operation: This is the largest part of all and contains all the routines for controlling the process and processing the data if necessary. The *Operation* sub-routine also includes the recovery actions for the workstation and therefore interacts a great deal with the *Outputs* and *Errors* sub-routines.

Only internal memory addresses are used here, no output is addressed directly.

Outputs: The workstation output routine contains all the output addresses and if the conditions are fulfilled, the outputs are activated. It also contains a set of 'last minute' protection against activation of a given output in critical cases.

Errors: In the event of an error occurring, the PLC program will jump to this routine. Each station has a general error bit which is set whenever an error occurs and allows the monitoring computer to know what is going on. This general error bit is triggered by individual error flags related to various parts or actions of the station. The general error, as well as the individual error that triggered it, is latched until the station is manually reset or the emergency stop is activated. This allows easy tracking of faults.

This routine also includes the recovery actions to be taken when a type of error occurs. There are three different types of error that can occur in the purpose built workstations: Status Error, System Error, Station Error.

An example of a recovery action would be when the filter appears to be blocked on the extraction workstation (no pressure rise), vacuum is switched off and positive pressure

is blown through the filter in an attempt to clear the blockage. The situation is then monitored for a few minutes and if no improvement is seen then an error will be flagged and the station will stop.

## 4.2. Soil Extraction Workstation

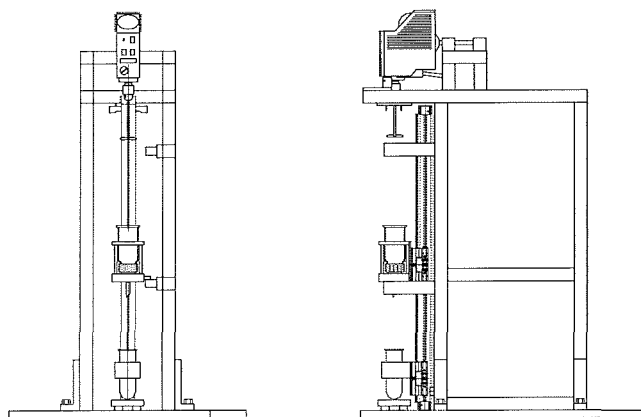
The Soil Extraction Workstation (SEW) is designed to extract and filter soil samples using solvents and an agitation mechanism. In the manual process, a solvent is added to the sample to cover the surface of the soil to a depth of about 3 or 4 cm. The sample + solvent is then placed on a shaker for approximately 20 mins, after which it is centrifuged and the solvent decanted off by hand. This process is then repeated a number of times.

### 4.2.1. Solvents

The design of the workstation allows the flexibility for the user to choose between different solvents. A study has demonstrated that 4 different solvents, most commonly used, would be required. However the solvent lines can be used with any compatible solvents.

The most commonly used solvents are: Acetonitrile, Acetone, Methanol and Water.

The solvents are dispensed to an accuracy of  $\pm 5$  ml or  $\pm 5\%$ , whichever is greater and the volume to be added is limited to 250 ml.



**Figure 19: Soil Extraction Workstation**

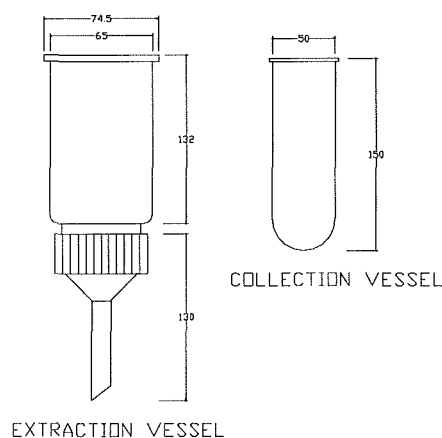
### 4.2.2. Mixing

The agitation method chosen is a stirring mechanism which is more suitable for automation than shaking. The solvent/sample contact time is the same. Particular attention was paid to the design of the paddle for the stirrer. This paddle had to cope

with the different types of soils to ensure good mixing between soil and solvent. The requirements were that the speed and the time of stirring can be set by the user. Stirring speeds include the range 500 rev/min - 1500 rev/min, though some higher speeds are used for cleaning purposes. Stirring times are in the range 2-30 minutes.

#### 4.2.3. Glassware

The extraction flask (250 ml) and the collection vessel (215 ml) used to recover the filtrate, were developed at the same time as the extraction/stirrer workstation. Figure 20 shows the agreed design/dimensions for those vessels with Soham Scientific.



**Figure 20: Glassware used in the SEW**

Particular attention was paid to properly seal the contact between extraction and collection vessels so that no losses of solvent occurred when the extract is passed through the filter.

#### 4.2.4. Filtering

Instead of centrifuging and decanting off the resulting sample, a process more suitable for automation was studied. If the sample is contained in a flask with a sintered glass bottom, then by applying vacuum or positive pressure the filtrate can easily be isolated. A pre-study revealed that an extraction flask fitted with a glass sinter or filter paper is ideal for the purpose. Coarse porosity filters (2 or 3) do not prevent solvent dripping through and a sinter of porosity 4 (10-16  $\mu\text{m}$ ) was giving the best results. However in the automated system, by applying positive pressure from under the sinter, it prevents solvent dripping even at porosity 1. Finer porosity sinters (3 or 4) may be blocked with some soil types (high clay); extensive testing has shown that porosity 2 is the best compromise.

The basic process for the SEW is as follows:

- Load extraction vessel containing soil sample
- Load collection vessel
- Put both vessels in contact
- Raise both vessels under the solvent dispensing nozzles and stirrer paddle
- Add a given volume of solvent (set by the user or super-user)
- Stir for a given time (set by user) at a given speed (set by user)
- Lower and unload the vessels

#### 4.2.5. Sub-systems integration

##### **4.2.5.1. Stirrer System**

The instrument used in this station is a Heidolph Model RZR 2102 laboratory stirrer. It contains a brushless DC motor drive with feedback of speed and torque. It has two speed ranges, 40-400 rev/min or 200-2000 rev/min. It is possible to switch between these ranges remotely. Only the upper range is currently used. The stirrer is controlled through an interface box which provides a 4-20 mA or 0-10 V control signal for the speed and torque. Two analogue modules were purchased for the PLC: A Digital to Analogue Converter (DAC) module and an Analogue to Digital Converter (ADC) module. Hence duplex communication would be established between the stirrer and the PLC. The stirrer speed is one of the parameters that is set by the user, but in normal use the speed would be set to the maximum that doesn't result in excessive splashing. The speed of the paddle is gradually increased during the stirring part of the cycle to prevent splashing. Feedback from the paddle speed sensor is used to detect if the paddle is stuck and not turning. The stirring time is controlled by a minute counter. After the required number of minutes the stirrer speed is ramped down and left turning slowly for the next phase of the cycle. If at any time an error is detected, the stirrer stops.

##### **4.2.5.2. Manifold System**

The manifold system allows to move the vessels from the loading/unloading position, to the processing position. The manifold is based around a Festo linear slide with two carriages and the necessary sensors for feedback on the carriages positioning.

The manifold system is set to work in three different modes:

- Initialization (solvent pumps are primed for a few seconds)
- Washing (a purpose built vessel is loaded to clean the stirrer paddle and other contaminated parts)

- Extraction (this is the normal processing mode)

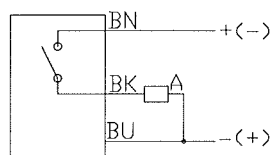
During extraction, the upper support holds the extraction vessel, while the collection vessel is loaded onto the lower carriage. Once both vessels have been loaded by the robot, the lower carriage joins the upper one at mid-stroke, and both supports are then raised under the solvent nozzles and stirrer paddle. A measured volume of solvent is then dispensed into the extraction flask while a slight positive pressure is allowed inside the collection vessel to limit the amount of solvent passing through the filter during solvent addition and mixing. After the soil and solvent mixture has been stirred for the required amount of time, vacuum is applied to the collection vessel to help pull solvent through the filter. Once the end point is detected, both vessels are lowered to their respective loading/unloading positions.

A similar approach is taken for the other modes except that only the upper carriage is occupied. In the case of the initialization mode, a look alike blanked extraction vessel is loaded and acts as a waste bottle to collect the solvent primed from all pumps.

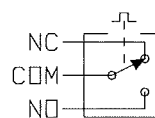
When the washing mode is selected, the upper carriage is loaded with a specially designed vessel connected to a sump and subsequently the drain. When this washing vessel is raised to the stirring head, some water is dispensed and the paddle is rinsed by spinning it at high speed. This will consequently induce a lot of splashing inside the wash vessel, allowing to rinse the top flange and dispensing nozzles at the same time. After a few seconds, the water is drained and replaced by some fresh one. This process is then repeated for three cycles after which the washing vessel is lowered to be unloaded.

The following sensors are used in the SEW for manifold positioning information:

- microswitches to confirm correct location of collection and extraction vessels
- magnetic limit switches to detect bottom position of lower carriage, upper position of lower carriage (wash mode), upper position of lower carriage (wash mode), and upper position of lower carriage (extraction mode)
- microswitches to detect lower position of upper carriage and upper position of upper carriage



Inductive Proximity Sensor



Spark Proof Micro-switch

**Figure 21: Limit sensors used in the Soil Extraction Workstation**

Spark proof limit switches are used where solvent vapours might be present. Even at low voltages and currents, a small spark might form as the contacts open.

#### 4.2.5.3. Vacuum System

After adding solvent and stirring, a valve opens to apply vacuum to the collection vessel to draw the solvent through the filter. This is the most critical step, as the filter may get blocked by the finer soil particles. Experience will determine the most suitable grade of filter paper for a particular soil type and the maximum level of water content for a given soil type and filter. Even with these precautions, the system must be able to detect if the filter is blocked and at the very least move the vessels out of the way, or preferably try alternative strategies to clear the blockage. The vacuum needs to be released before the vessels are lowered.

The main components used are a vacuum pump, a vacuum regulator, a 3/2 solenoid valve to connect up the vacuum line and vent to slight positive pressure, a vacuum sensor with 4-20 mA analogue output, and a solvent collection trap to collect any evaporated solvent. All the components of the vacuum system are solvent resistant.

The vacuum pump is a KNF Neuberger connected to a gas ballast and a solvent trap made by a condenser refrigerated by a chiller pump. The temperature of the condenser is maintained at approximately 5°C. The size of the flask used to collect the condensed solvent vapours has been calculated so that it allows continuous operation for about 7 days before having to empty it. For safety reasons, the user is required to check the solvent trap on a daily basis.

A steady vacuum of around 50mbar is produced to draw the solvent through the filter.

A solvent resistant solenoid valve connects the vacuum to the system. There are some solvent vapours present, and so the seals inside the valve must not expand due to the solvent, otherwise the valve would be liable to jam. The valve used is a 3-way valve to



allow the system to be vented to the atmosphere. The volume that must be evacuated is approximately 1 litre, and this is evacuated in less than 30 seconds. The flow rate through the valve is therefore 2 litres per minute.

Suitable materials for the valve would be stainless steel and Teflon. Teflon is sometimes not the best material for vacuum operations as it can be difficult to seal. However the distributors of a number of specialised laboratory valves were consulted and they concluded that there should be no problem in using Teflon seals with the levels of vacuum used here.

A 3-way solenoid valve from NR Research, distributed by Alpha Controls, was chosen. This gives a good flow rate of 8 litres/min at the design conditions.

A solvent resistant vacuum sensor is required, reading in the range 10 - 1000 mbar gauge pressure, with a 4-20 mA output. The sensor should also be able to cope with over-pressure, as an alternative technique is to add positive pressure to the top of the sinter to aid filtration. The sensor chosen was a Druck absolute pressure transmitter, Model PTX-520 with a range of 0-1.6 bar absolute, accuracy 0.3% FS (i.e. 5 mbar). The maximum over-pressure is 4.8 bar. All wetted parts of the sensor are made from stainless steel for complete resistance to all the solvents used. A stainless steel adapter connects the pressure sensor to the vacuum system.

The 4 - 20 mA output option was chosen because it is less liable to pick up interference from other equipment. The maximum and minimum readings are recorded continuously and a falsely high or low reading would severely affect the vacuum monitoring system. The current loop output is slower to respond to transient pressures than a voltage output, but interference will not cause voltage spikes.

Since the sensor gives a continuous readout, it is possible to cope with varying levels of vacuum via software. This would not have been possible with a simple or even a dual set point vacuum switch. The sensor allows the maximum vacuum level to be monitored and an error flagged if this is not sufficient. The pressure rise that signifies the end point is sensed relative to the maximum vacuum level. If necessary the sensitivity of the end

point detection can be adjusted, i.e. different levels of pressure increase can be used to trigger the end point signal.

#### **4.2.5.4. Fluidic System**

The solvent delivery system was developed so that the user can choose between several commonly used solvents. A mixture of up to three different solvents can be used for extraction. In the laboratory, scientists commonly use more than three solvents. On the SEW, this was made possible by fitting the station with pumps compatible with more than one solvent type.

The vertical height from the solvent storage to the dispensing position is approximately 2m.

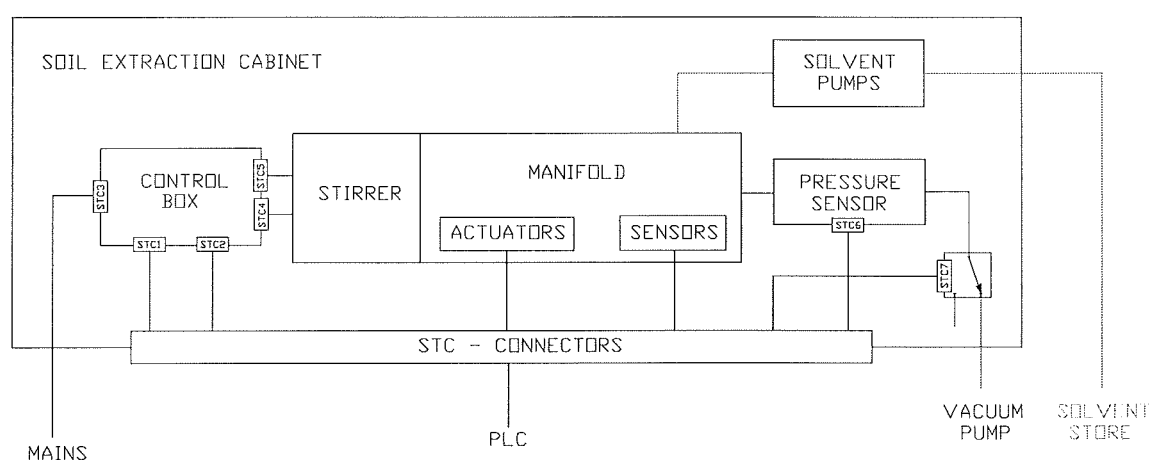
The pumps used are self priming, and although the metering accuracy is only 5%, this cut out a number of possible pumps designs. The most suitable type of pump found was a positive displacement pump. A diaphragm pump is particularly suitable as the only wetted parts are the diaphragm, check valves and ports. The pumps operate of a 24Vdc power supply, and deliver 100 ml in around 20 seconds (i.e. a flow rate of approximately 300 ml/min). The pumping pressure is approximately 0.5 bar (5 m head of water) in order to deliver a jet of solvent for cleaning the stirrer paddle.

The pumps are controlled via a timer on the PLC, which is calibrated to dispense the required quantity of solvent. The pumps operate on an intermittent duty cycle, averaging three operations per hour over 100 hours per week, with each operation lasting 20 seconds. The average duty is therefore approximately 90 hours per year.

A very important consideration when pumping flammable fluids is to avoid a build up of static charge, which could lead to a spark and fire risk. Normal practice when pumping flammable liquids is to use metallic pumps with all pump components grounded. Plastic can build up static charges but will not conduct and cannot be grounded (95-96 Cole Palmer catalogue p.1039).

For low volumes and intermittent use, it is possible to use a polymer pump head, and avoid the problem of static build up by passing the fluid through a grounded stainless steel nozzle before discharge.

The KNF diaphragm pump used is a low cost option. It consists of a fixed speed 24Vdc diaphragm pump, with polymer pump head. A variety of pump head options are available for compatibility with different fluids. The pumps cost around £120, depending on the material used in the pump head. Two different pump head materials are required to cope with the solvents to be used. The low flow rates involved does not lead to any significant risk of enough static charge building up to cause a spark. As an additional precaution, the solvent is passed through a grounded stainless steel tube before entering the extraction vessel.

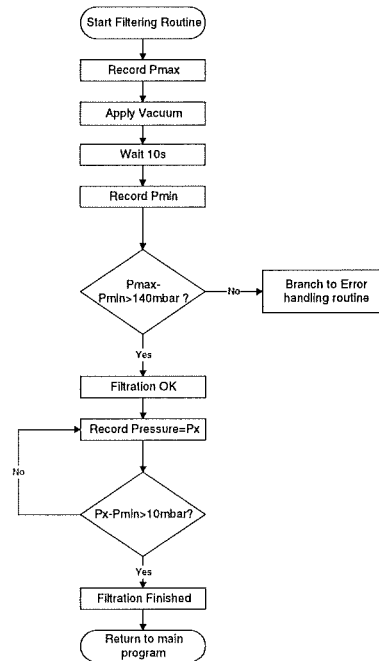


**Figure 22: Soil Extraction Workstation Connections**

#### 4.2.6. Control/Operational Strategy

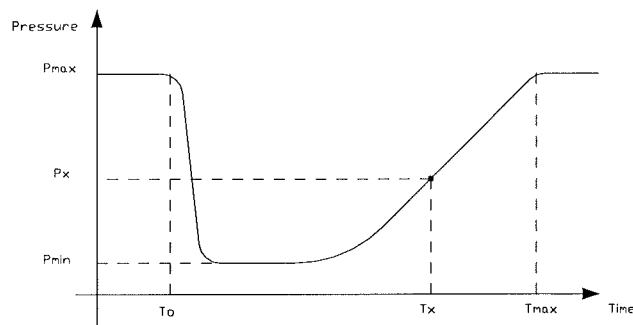
After the sample has been stirred for a set time, vacuum is applied to the bottom flask to help pull the solvent through the filter. A solvent resistant pressure sensor is used with a range of 0-16 bar absolute pressure and a 4-20mA output.

The sensor allows the maximum vacuum level to be monitored and an error flagged if this is not sufficient. The pressure rise that signifies the end point is sensed relative to the maximum vacuum level. If necessary the sensitivity of the end point detection can be adjusted, i.e. different levels of pressure increase can be used to trigger the end point signal.



**Figure 23: Filtration Process Simplified Algorithm**

The filtration routine branches out from the main program once the stirrer has mixed the sample for the required amount of time. The pressure is recorded as  $P_{max}$ . at time  $T_0$ , the vacuum is then applied and the pressure starts dropping. The pressure is updated continuously and recorded as  $P_{min}$ . After 10s, if the difference between  $P_{max}$  and  $P_{min}$  is less than 140mbar then a problem has occurred. The filtration finishes when the difference between the pressure  $P_x$  at a given time  $T < T_{max}$  and the minimum recorded pressure  $P_{min}$  is greater than 10mbar. The vessels are then lowered to the unloading position where the robot will take them away.



**Figure 24: Pressure variation during filtration**

$T_0$ : Time where vacuum is applied to the bottom flask.

$T_{max}$ : Maximum time allowed for filtration (30 min. adjustable)

$P_{max}$ : Maximum recorded pressure (before filtration)

$P_{min}$ : Minimum recorded pressure

### **4.3. Plant Extraction Workstation**

**Manual process:** The Plant Extraction Workstation (PEW) is designed to extract and filter plant samples using solvents and a combined shearing and cutting mechanism to homogenise the sample.

In the manual process, a solvent is added to the sample to soak the plant material to a depth of about 3 or 4 cm. The sample + solvent is then macerated for approximately 1-3mins, after which it is decanted into a filter funnel containing a filter paper with vacuum applied. This process is then repeated a number of times by physically transferring the substrate to the maceration flask and adding solvent, etc.

**Samples:** Initially the different plant types this station must cope with are:

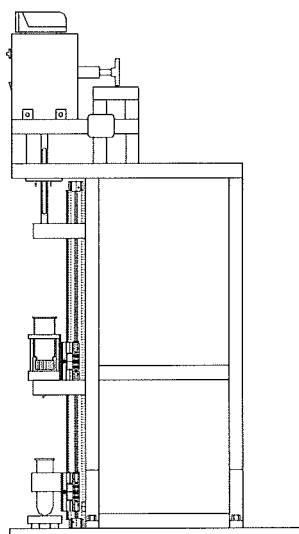
- Straw
- Grain

However ultimately any plant material may need to be processed, including high water content materials such as tomatoes and potatoes.

**Solvents:** The solvents used in this station are the same than in the SEW.

**Maceration:** The maceration process is performed by an overhead high-shear/cutting homogeniser. A pre-study evaluation showed that a Polytron PT6000 made by Kinematica AG was ideal for the process. Particular attention was paid to the design of the frame to accommodate the homogeniser. The requirements are that the speed and the time of maceration can be set by the user. Speeds of the homogeniser are in the range 0 rev/min- 24000 rev/min, although typically only up to 10000 rpm is used. Typical times are in the range 2-30 minutes.

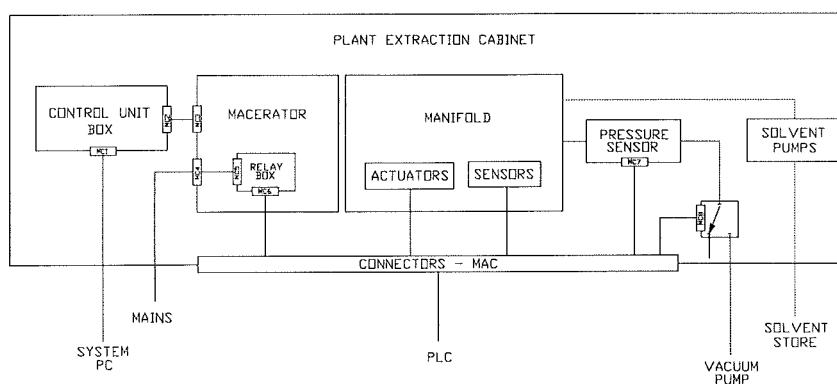
**Filtering:** The filtering system is the same as the SEW.



**Figure 25: Plant Extraction Workstation**

**Glassware:** The extraction flask (250 ml) and the collection vessel (215 ml) used to recover the filtrate are also identical to the ones used in the SEW.Soil Extraction Workstation

The macerator used in this station is a PT6000 Polytron Homogenizer with the RP502 Electronic Programming Unit. The speed can be adjusted from 0 rev/min to 24000 rev/min. The maximum noise level is 72 dBA at full speed. The optional RP502 Electronic Programming Unit allows the macerator to be controlled remotely via an RS232 link.



**Figure 26: Plant Extraction Workstation Connections**

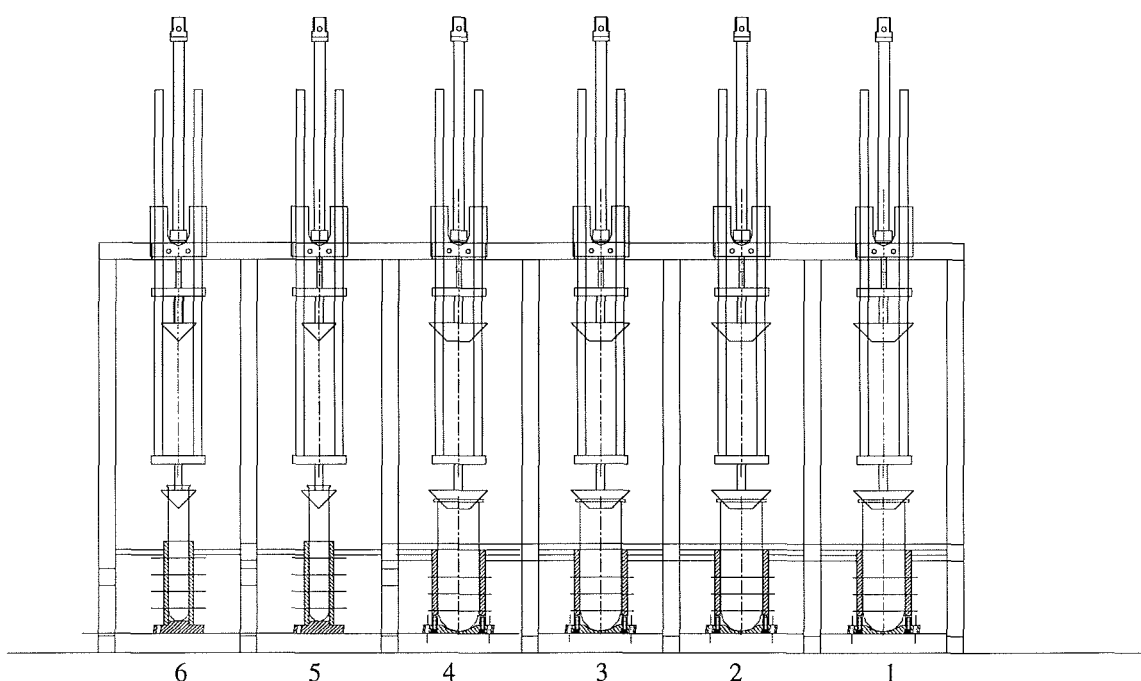
#### **4.4. Evaporation Workstation**

The Evaporation Workstation (EW) reduces the volume of sample by evaporating solvent. The end point of the evaporation is set remotely, to allow the user to decide

how much of the solvent to evaporate. A suitable off-the-shelf was not available on the market, so the workstation was custom designed. The closest commercially available device is the Zymark TurboVap® , which was unsuitable because of the unreliability of the end point sensing.

The Evaporation Workstation (EW) is split into 6 different modules: 4 modules for evaporation in 50 mm vessels and 2 modules for evaporation in 24 mm vessels. See Figure 27 below.

The EW consists of a rigid extruded aluminium frame that supports the different components of the station. Among them are the manifold system, the heating/temperature control system, and vacuum/compressed air system.



**Figure 27: Evaporation Workstation**

#### 4.4.1. General Operation

The user is required to enter two parameters via the GUI for this station to operate. The first parameter is the temperature at which the evaporation should be performed. Variable temperature settings are particularly important when dealing with solvents with a low flash and boiling point. In most of the cases, the station should be maintained at a temperature that allows fast evaporation without destroying the metabolites of interest. The second setting required by the user is the target volume for evaporating down to. There is a choice of four levels over the height of the test tubes. The test tube can either

be a general purpose test tube or a collection vessel. The levels correspond to fixed volumes for a given vessel type.

The station was designed so that the vessel is capped once placed in an aluminium block using a pneumatic cylinder with linear guide and bearings. An air inlet allows a small air flow inside the vessel when the vacuum is applied. The combination of heat, vacuum and small air flow is effective for fast evaporation. The air flow disturbs the surface of the fluid increasing its surface area and increasing the evaporation speed. The solvent vapours are then condensed in a solvent trap placed at the vacuum pump's output. As the solvent is evaporated off, the level of the fluid remaining inside the glass vessel decreases until it reaches the target level set by the user, where the process will stop. The vessel is then switched back to atmosphere for a few seconds before the pneumatic cylinder is raised freeing the glass vessel, ready to be unloaded by the robot.

#### 4.4.2. Detection mechanism

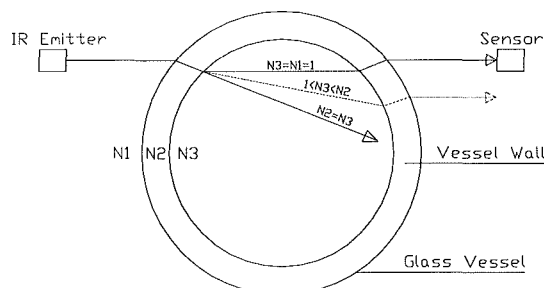
Each evaporator consists of an aluminium block featuring 4 level detection mechanisms spread over the height of the block. Each detection mechanism features a GL380 infra-red emitter and PT380F sensor.

The detection mechanism is based on a simple refraction principle governed by the refraction condition:

$$N1 \sin(I1) = N2 \sin(I2)$$

when a beam of light passes from media 1 into media 2.

- where:
- I1 is the angle the incident light makes with the normal to the surface.
  - I2 is the angle the refracted light makes with the normal to the surface.
  - N1 is the refraction index of the first media.
  - N2 is the refraction index of the second media.



**Figure 28: Evaporation End Point Detection Principle**

On Figure 28 above, the blue trajectory represents the path of light when refraction index  $N1=N3$ . This occurs when there is no solvent in front of the sensor. The red path



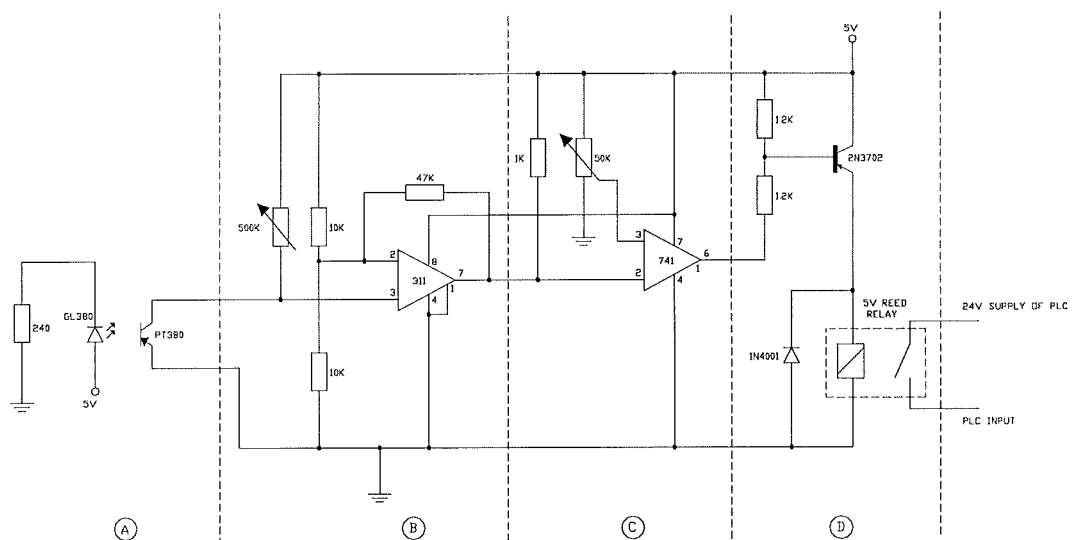
is followed when index N3 is that of a solvent, see Table 1 below. Solvent refraction indexes are all smaller than that of the vessel wall (Pyrex) but bigger than that of air, therefore the red path will always be below the blue path and above the black one which occurs when  $N_2=N_3$ .

In our case the following refraction indexes were considered:

**Table 1: Refraction Index of Solvents**

Medium	Refraction Index	Notation
Air	1	N1
Pyrex	1.474	N2
Methanol	1.326	N3
Acetonitrile	1.342	N3
Acetone	1.359	N3
Dichloromethane	1.424	N3
Water	1.333	N3

The detection system is split into four parts: A, B, C, and D. See Figure 29.



**Figure 29: End Point Detection Electronics Interface**

The detection part carried out in A features: an Infra-Red (IR) LED with current limiting resistor and an IR phototransistor. IR light is used limit the effect of ambient light. The output signal from the phototransistor is approximately 100 $\mu$ A which is too low to be picked up by the PLC. Therefore the signal needs to be amplified.

Circuit B acts as a Schmitt Trigger to switch its output between positive and negative saturated states when its input is equal to the reference voltage. The reference voltage for this circuit is 2.5V. This reference voltage can be altered by changing the two 10k $\Omega$  resistors.

C and D: The circuit action is such that the op-amp output is driven to negative saturation and the relay is driven on when the variable input voltage is greater than the reference voltage adjusted via the 50 k $\Omega$  potentiometer. The op-amp is driven to positive saturation and the relay is cut off when the variable input voltage is less than the reference voltage.

#### 4.4.3. Heating system

In order to speed up the evaporation process, the glass vessels are heated up by a mains powered rubber silicon jacket of 80W stuck on the outside of the aluminium block. Temperature feedback is obtained through a K type Nickel alloy thermocouple connected to an AD595 amplifier featuring an automatic cold junction compensation. The AD595 is a complete instrumentation amplifier and thermocouple cold junction compensator on a monolithic chip. It combines an ice point reference with a pre-calibrated amplifier to produce a high level (10mV/ $^{\circ}$ C) output directly from a thermocouple signal. The AD595 is powered from a single ended supply only (+5VDC) as negative temperatures are not measured. Given the range of temperatures, the thermocouple is required to measure: 20 $^{\circ}$ C (room temperature) up to 100 $^{\circ}$ C, the output of the thermocouple is in the range: 0.798 mV to 4.095 mV. The output of the AD595 is typically in the range: 200 mV to 1015 mV, which is then picked up by the PLC using an ADC module. The ADC input module can be configured to read an input range of 0 to 10V, 1 to 5V or 4 to 20mA, and -10V to +10V. The resolution is 1/4000 of full scale. Therefore, by choosing the range 0 to 10V, the resolution becomes: 2.5mV, which is more than enough to acquire with a good accuracy the signal from the AD595 output. The signal then needs to be scaled by software in order to be used in the program.

Based on the temperature of the block, the heating mats are turned on and off via a series of power relays. Each mat is supplied via a power relay and a security relay

common to all heating mats. The PLC outputs driving the relays are PID controlled using the PLC software.

The PID control function is a built-in function in the C200HS Omron PLC. The PID control algorithm provides better control of the temperature than would be possible using an on-off controller. There is a smaller temperature overshoot, allowing higher temperatures to be maintained without overheating.

Until the measured temperature is within 2.5°C of the desired temperature, the heater output is 100%. This brings the heating block up to near the desired setting. Within 2.5°C of the set value, the proportional term of the output is proportional to the difference between the set point and the present value of the temperature. PID control is carried out based on the specified input range of binary data from the contents of an input word. The proportional band is expressed as a percentage with respect to the total input range. With proportional operation, an offset (residual deviation) occurs, and the offset is reduced by making the proportional band smaller. If it is made too small, however, hunting will occur. Combining integral operation with proportional operation reduces the offset according to the time that has passed. The strength of the integral operation is indicated by the integral time, which is the time required for the integral operation amount to reach the same level as the proportional operation amount with respect to the step deviation. Proportional operation and integral operation both make corrections with respect to the control results, so there is inevitably a response delay. Derivative operation compensates for that drawback. The strength of the derivative operation is indicated by the derivative time, which is the time required for the derivative operation amount to reach the same level as the proportional operation amount with respect to the step deviation.

In our case the sampling period is set to 1s. The proportional band represents 2% of the total control range and integral and derivative operation have equal strength expressed by a constant set to 100. The heater output is Pulse Width Modulated (PWM). A counter is used to count 500 pulses of the built-in 0.02 second pulse clock. This counter is reset to produce a regular 10 second pulse. A second counter counts the number of 0.02 second pulses, counting down from the scaled output from the PID function, and is reset by the first counter. The heater is switched on when the status is “evaporate” and the second counter is off. Over each 10 second period, the heater is therefore on for the required percentage of the total time.

The first prototype which used this heating method used a 2 second pulse width but this was thought to cause excessive number of switching operations, shortening the life of the reed relay.

## **4.5. Vortex Mixer Workstation**

### 4.5.1. General

The purpose of the Vortex Mixer Workstation (VMW) is to mix the contents of vessels by rapidly rotating the base of the test tube whilst keeping the top still, thus forming a vortex inside the tube.

The VMW features two main sub-systems (see Figure 30):

- Manifold system
- Vortex mixers

The process on this station is as follows: the robot loads the vessel onto the corresponding mixer, the presence of the vessel is detected by a spark proof micro-switch. The process will start as soon as the robot is clear off the station providing that the required operating parameters have been received from the supervisory program. The operating parameters for this station are: mixing time for all three mixers and mixing speed for the larger vessel size mixer. If one of the parameters is missing then the process will not start and an error will be flagged to the supervisory program. Once all the parameters have been received, the manifold will be lowered onto the test tube and the mixer turned on. The mixer will stay on for a given amount of time set by the user. On finishing, the manifold will be raised after the mixer has been switched off.

### 4.5.2. Laboratory Instruments

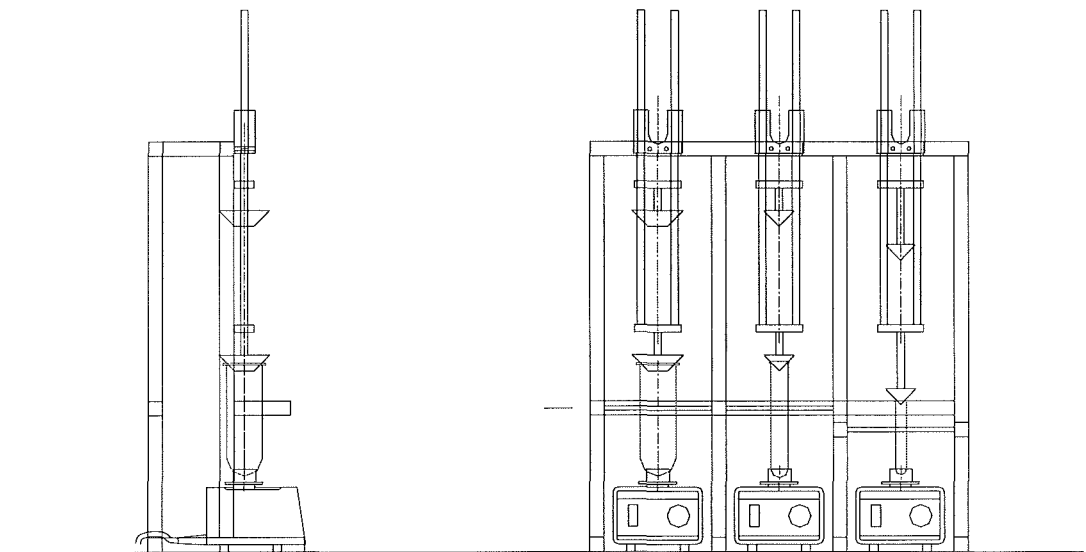
The station is based around three Heidolph laboratory vortex mixers, one for each of the three test tube sizes used in the system.

The vortex mixers are standard laboratory mixers from Heidolph Ltd (Model REAX 2000). The input power is 47 W. The shaking frequency of the split pole motor is 0 to 46 Hz, adjustable via phase control.

Similarly to other workstations, the VMW operating parameters such as mixing time and speed for the larger test tube size, are set remotely. Typical mixing time is between 1 and 2 min.

The mixer used for larger vessel sizes has been modified so that the speed can be adjusted remotely by the PLC based on the amount of solvent inside the vessel. This

allows to reduce splashing and ramp up the speed at start-up. The front panel potentiometer was replaced by 7 resistors of increasing resistance switched by the PLC via a series of reed relays. During operation, the PLC has been programmed to switch between the various resistors in ascending order until the set speed (or resistor) is reached.



**Figure 30: Vortex Mixer Workstation**

#### 4.5.3. Manifold System

The manifold system consists of an extruded aluminium frame as used throughout the cell. Three pneumatic cylinders with linear guide and bearing are used to cap and hold the test tubes in place while their content is being homogenised. The purpose of the manifold is to hold the top of each vessel in place while the bottom is being rotated by the mixer. An important consideration that had to be taken into account when integrating the mixers into the system is the force applied by the cylinder onto the mixer. Tests were carried out to find out what the best configuration was for the manifold and the vortex mixers. It was found that the manifold must be lowered to a point where it gets in contact with the top of the vessel but do not exert any pressure onto it. The design of a series of conic shape caps helped keep the test tube in place while allowing it to rotate during mixing.

The position of the manifold is therefore critical in the down position. Stop brackets have been fitted to the station to prevent the cylinder going too far down and some inductive sensors have been fitted to the pneumatic cylinder in order to get accurate feedback on its position. The type of sensors used is represented on Figure 31 below.

Spark proof and sealed sensors have been used wherever possible to avoid solvent incursion.

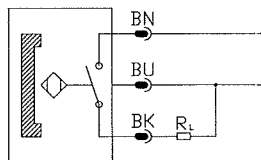


Figure 31: Inductive Sensor used in VMW

## 4.6. Ultrasonic Bath Workstation

The ultra sonic bath workstation USBW is used as an alternative homogeniser. This station has been designed so that it can also be used as a sample transfer workstation for transferring a sample from a test tube size to another.

### 4.6.1. Ultrasonic Bath System

The ultrasonic bath is a standard laboratory equipment that is used to homogenise the samples typically after evaporation or solvent addition. This station is also used to transfer a sample from one test tube size to another.

Three test tube sizes can be used in this workstation: 50mm vessel, 24mm and 16mm test tubes.

The laboratory bath has been modified so that it can be turned on and off remotely by the PLC via a power relay. A rack has also been made that allows all three tubes to emerge from the bath by the same distance, needed for the robot gripper clearance. The presence of the vessels is detected by sealed micro-switches (waterproof to IP 67) mounted above the water level but subject to splashing. The bath has also been fitted with a float sensor to control the level of the water inside the bath and refill automatically, using a 12VDC diaphragm pump, if the level drops below a certain level.

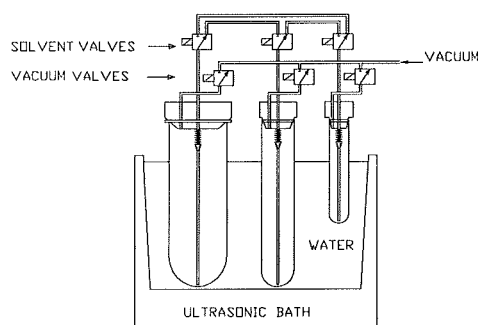
### 4.6.2. Manifold System

The manifold system consists of a pneumatic actuator and linear guide holding a support plate for the three caps necessary for the three test tubes. Each cap is fitted with a spring mounted needle the length of the inside height of the vessel it corresponds to. The spring allows the needle to touch the bottom of the tube without being forced down onto it, which may cause breakage. This limits to a minimum the volume of sample left behind during the transfer process. The needles and springs are made of stainless steel to avoid

corrosion. Caps and solvent tube connectors are all made of Teflon for solvent compatibility reasons.

#### 4.6.3. Fluidic and Air System

In order to transfer a sample from one test tube size to the next, a series of solvent and vacuum valves are required. Three vacuum lines are connected to the three vessels featured in this station. When a transfer occurs, vacuum is applied to the destination vessel through the activation of a solenoid valve. Vacuum is established for a few seconds inside the destination vessel before the solvent valves are opened to initiate the transfer. See Figure 32.



**Figure 32: Ultrasonic Bath Workstation Fluidic and Air System Connections**

The station is used either as an homogeniser or as a sample transfer workstation. In both cases the ultrasonic bath is turned on but in the first instance, the manifold is not lowered. After several tests, a maximum length of time has been measured for transferring a sample from a given vessel size to the next. This estimation is used to trigger the end of the operation and raise the manifold. Direct feedback is not provided here but performance can be estimated fairly accurately using the balance. Weights of empty vessels, and weights of vessels before and after passing through a given station are recorded. Hence, by comparison of the weights before and after the transfer and the weight of the empty tube, it is possible to determine the amount of sample actually transferred into the destination vessel and take the appropriate decision for the rest of the process and inform the user of what is going on.

### 4.7. Centrifuge Balancing Workstation

#### 4.7.1. General

The Centrifuge Balancing Workstation (CBW) is a station internal to the system. The user do not need to input any parameter to run this station. This station is only used in

conjunction with the centrifuge and its purpose is to dispense a mass of water equal to that of the sample to be centrifuged. The 6K10 centrifuge used in the system (see section 3.8) features an automatic imbalance detection system. The tolerance for imbalance is 10 g for opposite buckets and 100 g for adjacent buckets. Therefore, by placing a sample in one of the buckets, an equal weight  $\pm 10$  g must be placed in the opposite one.

Before running the centrifuge, the robot will go through a centrifuge balancing process. It will weigh and place the sample into one of the buckets of the centrifuge. An empty vessel of the same type is then loaded onto the balance of the CBW. A given amount of water is then dispensed into the vessel by a peristaltic pump until the target weight is reached (weight of the sample). The robot then unloads the CBW and loads the opposite bucket to the sample in the centrifuge with the counterweight vessel.

#### 4.7.2. Manifold System

The manifold system consists of an aluminium frame and a pneumatic cylinder with the required control valve, pressure regulators and connectors. The cylinder is fitted with a dispensing needle connected to the peristaltic pump which is moved back and forth above a glass vessel sitting on a balance. The cylinder is extended above the vessel only when dispensing occurs and must be retracted when the robot is loading/unloading the station.

#### 4.7.3. Water Dispensing System

The dispensing of water is achieved with a Gilson minipuls 3 peristaltic pump interfaced to the system using input/output control connector situated at the back of the instrument. This 6-pin barrier strip connector gives access to two contact inputs and one analogue input to the peristaltic pump. The two TTL inputs have a threshold of 2 V and are pulled up to 5 V with a high impedance. They are protected against AC voltages up to 264Vrms for an unlimited period of time. The pump starts by closing the "start/stop" input and stops by opening it. The input is closed by shorting pin3 to pin4 (ground). When the pump is activated using this input, keypad commands have no effect until the "start/stop" contact is opened again. The start/stop control of the pump is achieved by the PLC via a 24 V reed relay.

Water is dispensed continuously by the pump until the target weight is reached.

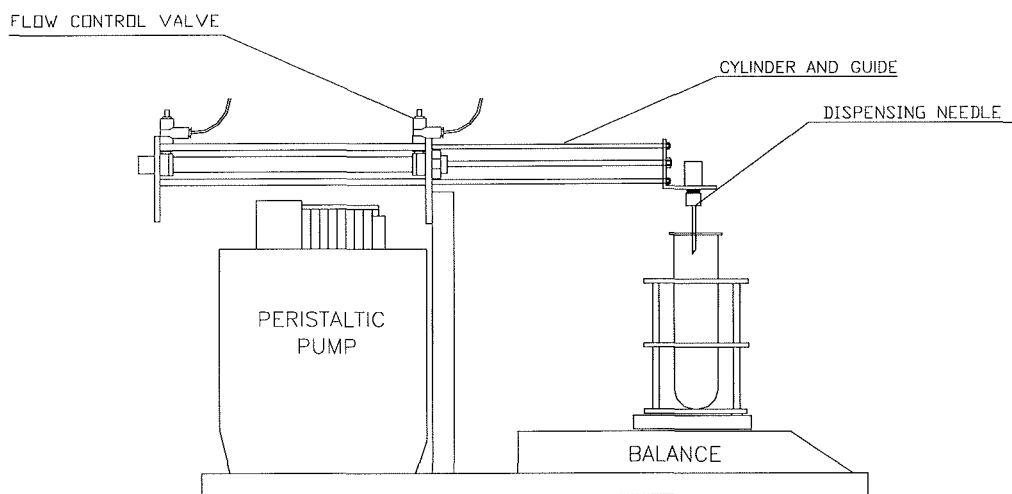
#### 4.7.4. Weighing System

The CBW also features a one decimal place Mettler balance model PE400. Unfortunately, this model no longer exists on the market and the one available at RPAL



did not feature any communication interface. The supplier was contacted and after several weeks provided us with a compatible communication card for the balance. The interface supplied was a uni-directional RS232 interface card with no transmission interruption. Therefore, the balance had to be connected to the PLC which allowed to activate remotely an interruption switch through a relay interface, so that the balance would only transmit when requested. The supervisory program sends a request to the balance every other second to transmit the weight.

The dispensing rate of the pump is approximately 21 ml/min. Therefore, during two seconds (weight reading intervals), the pump dispenses 0.68 ml of water or 0.68g which is the maximum possible error between two readings. This is perfectly acceptable as the centrifuge can accept up to 10g.



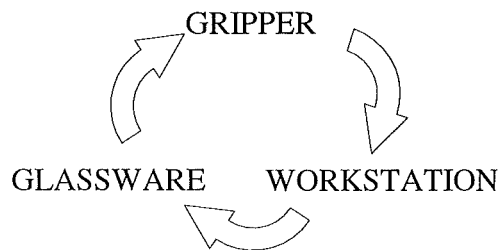
**Figure 33: Centrifuge Balancing Workstation Layout**

## 5. OVERALL SYSTEM INTEGRATION

### 5.1. General

All twenty two workstations around this system have only two things in common: the robot gripper and the glassware. The integration will be successful only if the emphasis is put on these common interfacing components but all three entities have to evolve in parallel. See Figure 34

Design/Integration cycle:



**Figure 34: Design Cycle**

As part of the integration of the purpose built workstations, some instrument drivers were written in Delphi

### 5.2. Manipulator and track

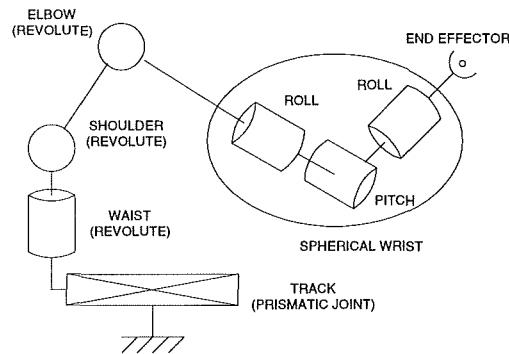
#### 5.2.1. General

The choice of the robot played an important part in integrating many different instruments as it is the key to mechanically interface all those instruments together.

Robots designed specifically for laboratory applications have limitations over small general purpose industrial robots. The Zymark robot is a cylindrical robot with four degrees of freedom (DoF), and although suitable for many applications, it cannot easily be mounted on a track. Between 1988 and 1994 Isenhour (1989 and 1991) and his group have reported the use of expert system software to control robotic systems using the Zymark robot. The Hewlett-Packard ORCA robots promised much, with its superior software and control capabilities (Owens, 1989), (Bleyberg, 1990). However, the lack of a waist, small pay-load (0.5 kg), and limitations on track length (2 m) again made it unsuitable for our application.

The chosen manipulator is a CRS A465 6 Degrees Of Freedom (DOF) Manipulator with a similar kinematics representation to the KUKA 6/1 or PUMA 6-axis, see Figure 35 below. This robot has also been used successfully for other laboratory automation

applications (Cockburn-Price, 1995 and Ogden, 1996) most notable by North West Water, and is a prime component of Robocon systems.



**Figure 35: Kinematics of the CRS manipulator**

For improved mobility the manipulator is mounted on a 6 metre track. This allows to have only one manipulator servicing all workstations instead of having several inter-linked robotic cells. The system is therefore more compact and easier to control but robot travelling and handling times must be kept to a minimum.

The necessity for the robot to hold test tubes in a vertical orientation at all times has put the emphasis on avoiding the 6 sets of singularities of this manipulator when teaching locations and via points.

The CRS A465 is operated by a C-500 robot controller fitted with a SYStem Input/Output (SYSIO) module and a General Purpose Input/Output (GPIO) module. These two modules allow the robot to receive and generate signals directly to and from the PLC or other workstations without necessarily going through its host computer. The SYSIO module also allows greater integration of the emergency daisy chain of the robot into the overall system circuit.

### 5.2.2. Operation

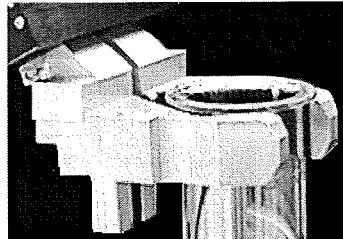
The robot routines are programmed in RAPL II and stored in the robot controller. The various routines are run remotely by a host computer through Dynamic Data Exchange (DDE) protocol commands. The robot is used as a pick-and-place manipulator. The pick-and-place locations are specified by the supervisory program based on the rack or workstation to be attended. During the process, the robot program keeps track of the occupied/unoccupied locations in racks and workstations as an additional security. As the process progresses, the robot program sets and resets location indexes. At the beginning of each run, the robot locations are reset to the initial state which is: all racks

are assumed to be full of vessels and all workstations empty. If by mistake, the robot is sent by the supervisory program to place a vessel in an already occupied rack location, the robot will flag an error to the upper hierarchical level preventing it to crash into an occupied location. During normal operation, the robot is issued with a pick-and-place set of locations, on completing the command, the robot goes back to a safe position in the middle of the track and sets an output to 1. This output is wired into the PLC and is used to trigger the start of the process for the workstations. This gains time in bypassing the supervisory program and sending the start signal directly to the PLC.

### **5.3. Gripper design**

The design of the gripper is one of the most important tasks when integrating a manipulator. The gripper is designed so that it fits in all the current workstations, but future stations will have to be designed around the gripper. Therefore the design is robust and simple, and the number of robot re-orientations is kept to a minimum. A single hand rather than multiple hands is chosen to reduce lost time in hand changing mechanisms.

A servo gripper fitted with two angled fingers is used because its ability to control finger position makes it suitable to manipulate objects of varying size. A force sensor is integrated into the servo gripper so that real time feedback is obtained on the state of the fingers.



**Figure 36: Robot Gripper**

The servo gripper, supplied with the robot, had a maximum opening distance of 50 mm, but the vessel diameters ranged from 12 to 70 mm. In order to overcome the problem of lifting the larger vessels, the gripper hand had to be open by a least 20 mm. This made it impossible to lift the smaller vessels, so two lifting positions were required on the same hand. Another solution was to use inter-changeable gripper hands, but the additional cost, and time delays, in switching hands during use did not look an attractive proposition, so considerable effort was put into the gripper design. Prototypes were made out of wood in order to obtain a satisfactory working gripper before the final design was machined in aluminium. The design evolved over a period, and was

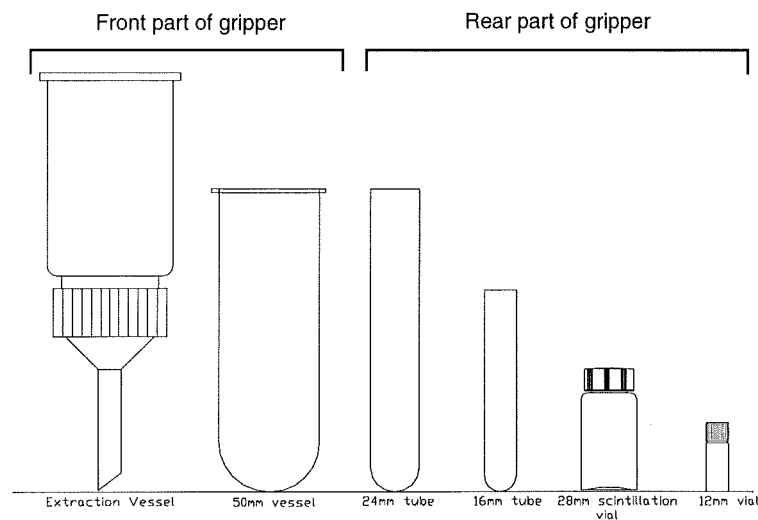
orientated at an angle of 25° from the vertical, in order to achieve maximum vertical lift, which was required for the extraction workstation. In addition, rubber pads were added to protect vessels from damage by the aluminium. The front part of the gripper handles the large vessels, the rear the remaining smaller vessels.

The design of the gripper shown above allows to grip a wide range of vessels and calibration weights with an outside diameter in the range: 11.5-65 mm.

A 4-point grasping restraint is applied to any test tube for accessibility and implementation purposes. A 2-point restraint was not enough to stop the glass vessels from tilting during robot displacements and a 3-point restraint was thought to be too complex and costly to implement.

#### **5.4. Glassware**

The vessels were optimised for the system, during the design stage, but the selection was, wherever possible, based on standard laboratory glassware. A total of six vessels were selected, based on the volumes of sample extracts used, and to optimise the transfer of aliquots. The extraction vessel is the initial vessel, containing the sample, and consists of a modified Schott Buchner funnel. The lower part is a polypropylene base funnel that holds a slotted polypropylene disc sandwiched between two solvent resistant seals, and secured with a screw-in Pyrex glass filter head. On top of the slotted disc is placed a suitable glass-fibre filter disc that is held in place with a stainless steel mesh. The soil or plant material is then added prior to running on the system. Three intermediate vessels are used in the system for the bulk of the sample work-up, and consist of carefully selected vessels enabling the minimum number of vessels, yet allowing the maximum flexibility of extract manipulation. The volume ratios between consecutive vessels are approximately five to one, allowing between 200 ml and 8 ml of solvent to be manipulated at the two thirds full mark. These tubes are 50 x 150 mm, 24 x 150 mm, and 16x100 mm, the last two being standard test-tubes. Final extract vessels are the LSC vial, and a standard 12 mm vial suitable for most automated chromatographic instruments (GC or HPLC). On-line GC and HPLC were incorporated into the design, and is the next phase of the project. The final vessels selected are shown in Figure 37.



**Figure 37: Glassware**

Optimisation of the shape and size of the vessels went a long way towards finalising of the gripper design, the final vessels being cylindrical in shape. This enables the robot gripper to grip the vessel, regardless of size in the same manner. The vessels were, wherever possible, selected with a round-bottom as accuracy became less of an issue. The round-bottom aids placing by guiding the vessel into the support rack during the placing operation, whereas a flat-bottom vessel needs to be accurately placed, and the rack location bevelled.

In addition, the larger, heavier, vessels were designed with a lip to aid robot lifting.

### **5.5. System Power Supplies**

The power supply is also common to all workstations. There are three different circuits supplying the automated system:

- Normal Circuit (rated: 32A max.)
- Maintained Circuit (rated: 32A max.)
- Un-interruptible Power Supply (UPS) Circuit (rated: 32A max.)

The reason for having three different supplies is the level of criticality of each instruments. If a power failure occurs, it is vital that workstations where a change in volume occurs during the process are sustained.

Workstations where no change in sample volume occurs are connected to the maintained or normal supply i.e.: the operation can be interrupted and repeated later.

Workstations where a change in volume occurs, are connected to the UPS so that there is no break in the power supply.

When a power cut is detected, the UPS will notify the overall system control program, and monitor the mains. If power is not re-established after a given time (set by super-users), the UPS will run a shut-down routine that will override the controlling program and close and shut-down all the computers. The routine consists simply in leaving the system in a safe configuration before turning the power off. During this routine, the robot will empty of glassware as many workstations as possible except for the critical ones still in operation. Most of the system instruments will then be turned off allowing the UPS to sustain the strict minimum of stations still in operation until the UPS battery low signal where everything is turned off. The UPS specified for the system is rated 8kW and, with the current load, can sustain all the workstations running at the same time for 30 mins which is enough time for most of the processes to finish.

Another benefit of the UPS is that it acts as a filter to prevent spikes or brownouts from affecting the computers and other instruments.

## 6. IMPROVED OPERATIONAL STRATEGY

An important factor to achieve increased capacity is the use of semi-autonomous workstations instead of devices that require the robot to work them (Little, 1993). The 'stand-alone' modules operate on their own so that the robot can carry out other functions. In this way, the robot becomes a 'pick-and-place' manipulator, whose only function is to transfer sample containers between workstations where the different operations are performed. The result is that many procedures can occur simultaneously on samples at different stages of a procedure. However these stand-alone modules are not independent. Their performance has to be synchronised and controlled by a host computer.

The RPAL system integrates twenty two workstations. Some of them are off-the-shelf serial devices fitted with bi-directional RS-232 interfaces which can be connected directly to a computer. However, older instruments or custom built workstations, do not have RS-232. These workstations are instead controlled through digital or/and analogue signals. Data acquisition control cards, Programmable Logic Controller (PLC) and micro-controllers, were the interface options studied.

A Sysmac C200H PLC (Omron) was chosen based on the high number of I/O and timers required. The need for analogue control signals, and other factors such as cost, development time and expandability were also considered. As this PLC is fitted with an RS-232 interface, it can be easily integrated into the system.

### **6.1. Manipulator control & operation**

In order to limit communications between the host computer and the robot controller, the pick-and-place set of data is condensed in two strings of data, one for the pick and one for the place location. The data is organised in various variables arranged in strings and decoded by the robot controller. The variables are %1, %2, %3, %4, %5, %6, and %7.

%1: Safe Location on track

%2: Intermediate Position

%3: Approach Position

%4: Final Location

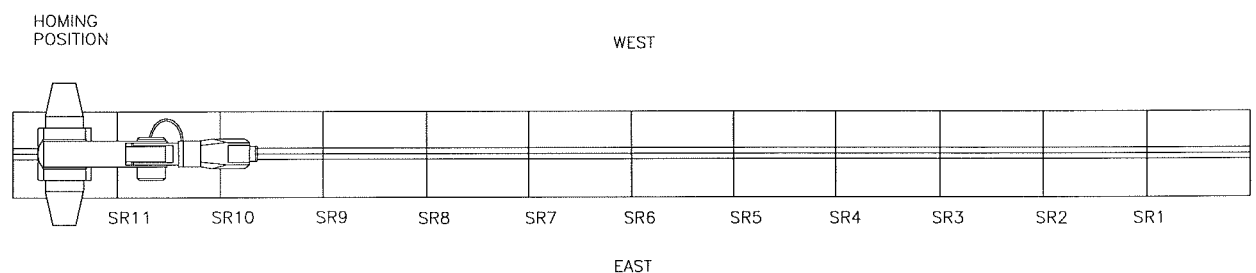


%5: Safe distance above final location

%6: Lipped vessel or not (specific gripping routine)

%7: Terraced rack or not (specific approach routine)

The concept of safe positions is used to ensure that the robot moves down the track in a safe and predictable manner. These safe positions are with the robot arm and the gripper in a horizontal configuration, see Figure 38. These safe positions enable the robot to travel between them without collision with equipment. Starting from the opposite end to the homing position the track has been divided into twelve 0.5 metre sections, numbered 1 to 12. The locations are called SR1 to SR12, S standing for Safe, see Figure 38 below. The variable %1 is used within programs to update the safe location. The track is then divided into East and West so that a location can be allocated to one side of the track or another.



**Figure 38: Safe positions on robot track**

#### Rack Locations:

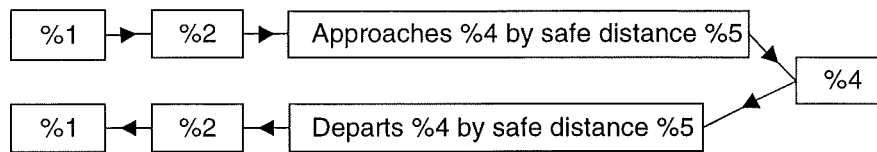
Storage racks for vessels are approximately 500 x 250 mm in size and contain a number of vessels arranged in a grid.

Each storage rack is associated with a vessel type, and a number of rack positions within the rack area, such that the locations are stored in an array (with R standing for rack). R5E50[001] is therefore rack 5, east, for 50 mm vessels, position number 1.

### **6.2. Picking and Placing Vessels**

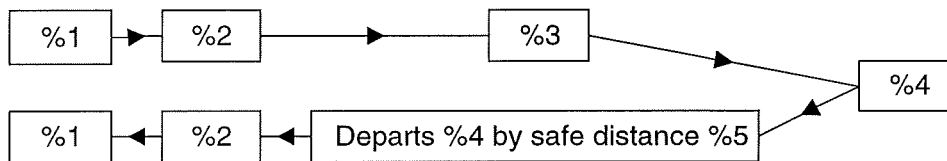
In order to pick up a vessel the robot (starting from the safe position defined above) must negotiate a safe route to the vessel in question, pick it up and return to the safe position by the same route. Similarly when placing a vessel the robot must negotiate a safe route between the safe position and the destination. In order to do this several

intermediate points on its route to the final destination are defined. The variable parameters %2 (intermediate position), and %5 (safe distance above the destination) are used together with %4 for the final destination. Mixing these variables up between routines is liable to be disastrous and so the positions must be thoroughly checked before running the routine. A test routine using slower speeds is used to test the procedure before running it at full speed. The routine thus goes in sequence:

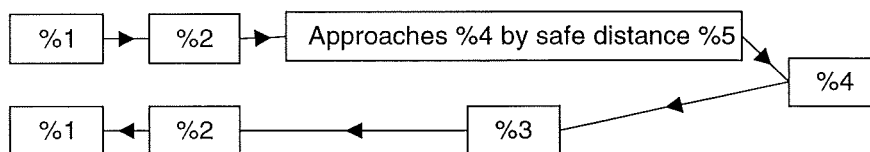


**Figure 39: Pick-and-place sequence for normal racks**

In order to pick a vessel from a terraced rack, approach from the side, and departure vertically is required, and the reverse to place a vessel. This variable is defined as %3. See Figure 40 & Figure 41.



**Figure 40: Pick sequence for terraced racks**



**Figure 41: Place sequence for terraced racks**

Gripping lipped vessels:

In the case of a lipped vessel, the robot goes under the lip and closes the gripper with a force so that the force sensor detects the presence of the vessel. The gripper then relaxes so that when it departs vertically by 10 mm, the vessel slides down the gripper, the lip resting on the top of the gripper. A final force is then applied in order to securely grip the vessel.

### **6.3. Cell Layout and Process Optimisation**

A graphical simulation model of the robotic cell was generated using AT&T Istel's 'Witness' Visual Interactive Simulation software. Visual feedback from the animated display provides process insight and aids in finding more effective workstation arrangements within the cell, reducing cycle time and allowing increased throughput and capacity (Smartt, 1997). The simulation model of the system was developed by initially modelling the workstations as independent "machine elements", which are standard components of the Witness package. The definition of the workstation element includes information such as estimates for the process time and variability, plus any special features, e.g.: producing extra aliquots, combining or transferring samples, etc. The workstations are represented on the animated display by custom drawn icons which change colour to indicate the status of the stations (busy, idle, error, etc.).

The robot track is modelled as a one dimensional array of sixty elements, with each element corresponding to a physical address within the cell. The estimated transfer time between adjacent workstations will therefore be of a similar magnitude to that between workstations on opposite sides of the track. This assumption is valid since the robot moves to a central safe position between each pick/place operation.

The model is animated by means of custom written functions which control the operation of the simulated system. Status updating functions are called as workstations begin or end a process and monitoring functions examine the status of each workstation and place calls to the robot as necessary.

At the beginning of each simulation run, each workstation is allocated to a particular address in the cell. The address reference acts as a pointer to the element of the track array corresponding to the workstation location. Calls are made to the robot when a workstation has finished its cycle using the pointer references, which are decoded to determine which element of the track array the robot should move to.

#### **6.3.1. Process Modelling**

Since the proposed system will operate with a high level of flexibility and no process may ever be repeated, it is not an easy question to determine which process to simulate in order to compare alternatives on an equal basis. If the results of the simulation study are to be valid, it is important that the simulated process is representative of the

processes likely to be performed by the actual work-cell. The final users and the personnel carrying out the operations manually were consulted, and decisions were taken as to the anticipated workload of the system.

Layouts were evaluated using a standard experiment which was considered to be representative. This consisted of a batch of four samples, with each sample subsequently being divided into three extracts. Further aliquots were taken from each extract through the system in a manner representative of the current manual process.

### 6.3.2. Robot Scheduling (Task Planning)

The proposed system is fully multi-tasking and many of the processes are non-deterministic, with a probability function defining the process time. For efficient operation, the robot is scheduled dynamically on-line. A status monitoring routine runs every minute, examining the state of each workstation and using a set of rules to determine whether a call can be made to the robot. A flag for each workstation is set to indicate when a call has been made, preventing duplicate calls. This flag is reset when the robot has completely finished the transfer task. In this study, the bottlenecks tended to shift as the process advanced, so all calls are given an equal priority and are answered in the order in which they were placed. More complex scheduling algorithms could be developed to further improve cycle times once the layout of the cell has been finalised.

### 6.3.3. Evaluation of Layouts

Correct selection of relevant success criteria is essential in quantifying the performance of the particular layout under investigation. The cycle time for the representative batch of samples was selected as the primary measure for evaluating layouts, and secondary measures were used to provide process insight. These included the total distance travelled by the robot, the time taken to answer calls and the percentage busy and idle times for key workstations during certain phases of the process. Stopwatch functions were used to investigate specific aspects of the model, for example the time taken to answer calls to bottleneck workstations.

The method of Closeness Ratings was used to determine the initial layout of the work-cell (Krajewski, 1990). A tally is kept of the number of transfers between each combination of workstations, and pairs of stations with frequent transfers are placed close together. It was decided that there should be a generally linear flow of parts

through the system from one end to the other, and this determined the approximate positions of the start and end workstations in the process. Workstations that were employed frequently throughout the process, for example the analytical balance, were initially placed towards the centre of the cell.

#### 6.3.4. Parameters to Vary to Reduce Cycle Time

Duplication of bottleneck workstations is one obvious method of increasing the system throughput (Goldratt, 1993). In addition, changes to the way some operations are carried out had a very significant effect on the performance of the system. Prioritising the bottleneck workstations had a minor effect. The use of extra storage capacity in the system also affected the throughput by allowing the robot to move samples away from bottleneck workstations whilst the next workstation in the process is still busy.

Key factors such as critical process times are varied to ensure that the model is not over-tuned for the particular process chosen to represent the system workload. This ensures that the solution is robust and performs well under a wide range of conditions (Dugendre, 1998).

#### 6.3.5. Results

The main bottleneck in the process was the robot, which was busy for over 50% of the time. The distance travelled by the robot during the test run was 1200m, with a process time of 24 hours 56 minutes. Measures therefore had to be taken to reduce the workload on the robot, and the effort was concentrated on reducing the transfer time to and from the frequently used workstations by rearranging the layout of the work-cell. These measures resulted in a 27% reduction in the predicted total process time to 18 hours 16 minutes.

A further result was the importance of redesigning the process itself in order to reduce the overall process time. Techniques that are inefficient and time consuming in terms of man power when carried out manually are also an inefficient use of system resources if automated directly. If possible, the process should be redesigned to make most effective use of available resources, whether these be personnel or machinery. For example, one particular technique known as liquid-liquid extraction is time consuming when carried out manually and in the simulated automated system this technique also demands a great

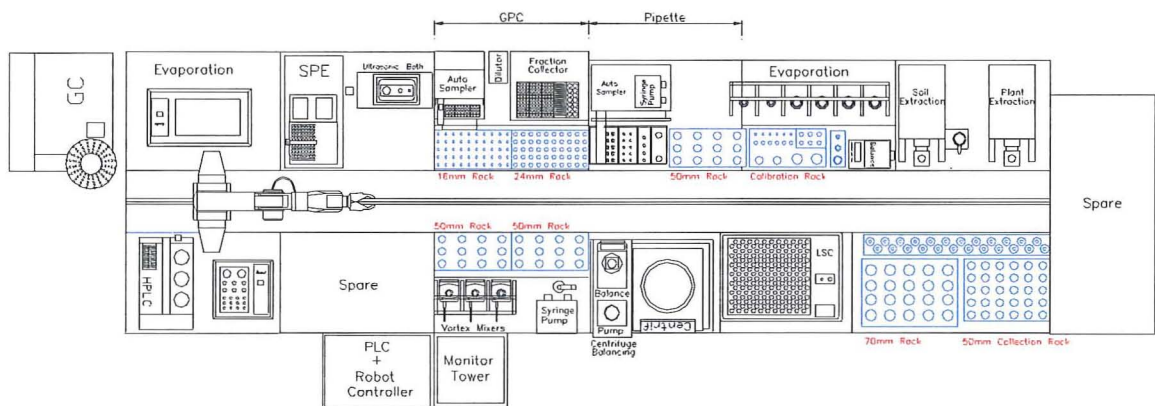
out manually and in the simulated automated system this technique also demands a great deal of system time. Since the technique is inherently inefficient, it should only be carried out if alternative methods are not appropriate.

Changes to the way the process was carried out resulted in more significant improvements than those achieved by changing the work-cell layout. It is predicted that if implemented in full, these changes would result in a 68% reduction in the representative batch process time.

In fixed based robot cells, reachable workspace is at a premium and geometric constraints often govern the location of workstations. The location of a robot within a work-cell is often determined by reachability and mobility criteria.

The more open arrangement of a track mounted robot system alleviates the shortage of accessible workspace inherent in fixed based robots and provides scope for future expansion.

See optimised cell layout in Figure 42 and in appendix.

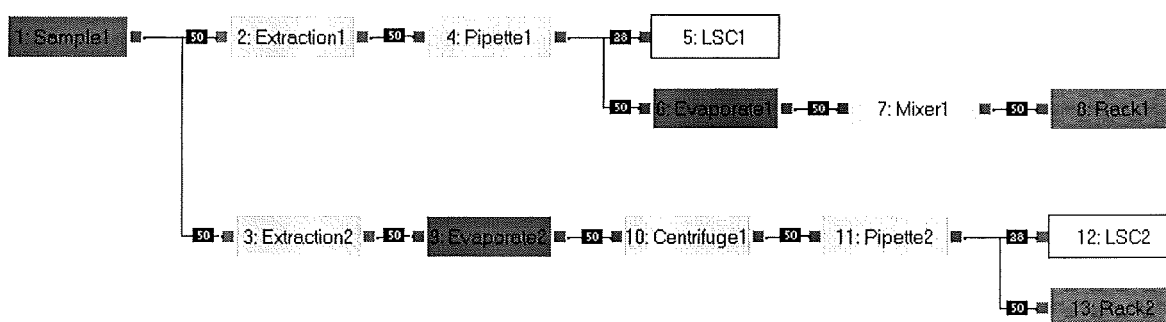


**Figure 42: Optimised Cell Layout**

## **6.4. System operation**

### **6.4.1. General**

The first step to operate the system is to design a method using the GUI. A simple drag and drop operation is required to add workstations to the method. Each workstation must be linked to the next and configured as required in order to complete the process.



**Figure 43: Method Designed using the GUI**

At design time, the GUI ensures that two consecutive workstations are compatible with each other by selecting automatically the correct vessel size for the operation. From the method input by the operator, the system calculates the resources needed and issues a mapping for loading the samples on the racks. The process will then start once an instrument calibration routine has taken place.

When a workstation is selected for operation by the scheduler, the pick and place parameters are downloaded to the robot controller with the command to move. Workstation running parameters entered by the user on the GUI are transferred to the selected instrument device driver via DDE (Dynamic Data Exchange) before the manipulator reaches it. The workstation then awaits two start signals to begin its task.

The first start signal will come from the computer confirming that all parameters are downloaded successfully; the second signal will come directly from the robot controller once the robot has loaded the station and has reached the nearest safe position. Once the station has finished processing a sample, it sets a call in a given run-time database notifying the robot application that the unloading process can take place. Workstations are prioritised by the robot scheduler according to their availability and throughput.

Each sample and its subsequent extracts are processed through the method entered by the user until it terminates on the rack where it was initially placed. If other subsequent products of the original sample are to be collected from other racks, a mapping of the locations is issued to the user.

#### 6.4.2. Graphical User Interface (GUI)

Barnett described the principle of the user interface as long ago as 1988. The off-line Graphical User Interface allows analysts, with minimal training, to develop a method via the computer screen. All workstations are represented graphically, giving the user complete flexibility for the analysis. Samples can be split into aliquots and processed

with any of the analytical techniques that have been integrated into the system. Methods are built by dropping workstations in the desktop, configuring them, and joining them to previous stations. The GUI interacts with the user in order to guide the analyst through the method design process. The system checks sample volumes, vessel compatibility and workstation parameters at every step of the process. This ensures that only feasible methods are stored for running. The method development and system set-up capabilities of the interface also contain an integrated reporting tool that generates formatted reports containing all the data, after the method is run.

### 6.4.3. Database System

The Database system is more than a data storage tool because it has control purposes and maintains GLP. It consists of a net of databases distributed in three sets. The first one, the “User-time” set, is functional at the design stage. These permanent databases store all the information related to methods designed with the GUI, such as user, station parameters and connections. The temporary “Run-time” set is used by the control programs. When a method is run, its related information is transferred from the User-time databases to the run-time ones. Controlling computers use these databases firstly as a source of information to know which operational parameters to download each time and also, to synchronise and schedule instruments and robot operations. In addition, on-line generated data, in the form of weights, times, errors, etc. are stored here. Once the process is finished, all the relevant data from the run-time set is transferred to a permanent set of “post-run” databases. These are exclusively related to the particular run of that method and are used for reporting purposes.

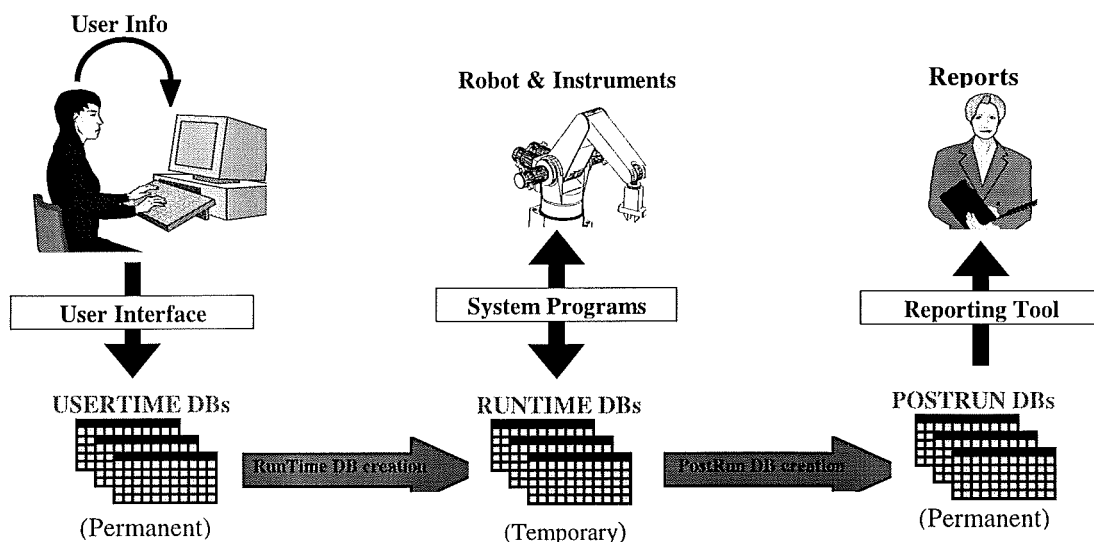
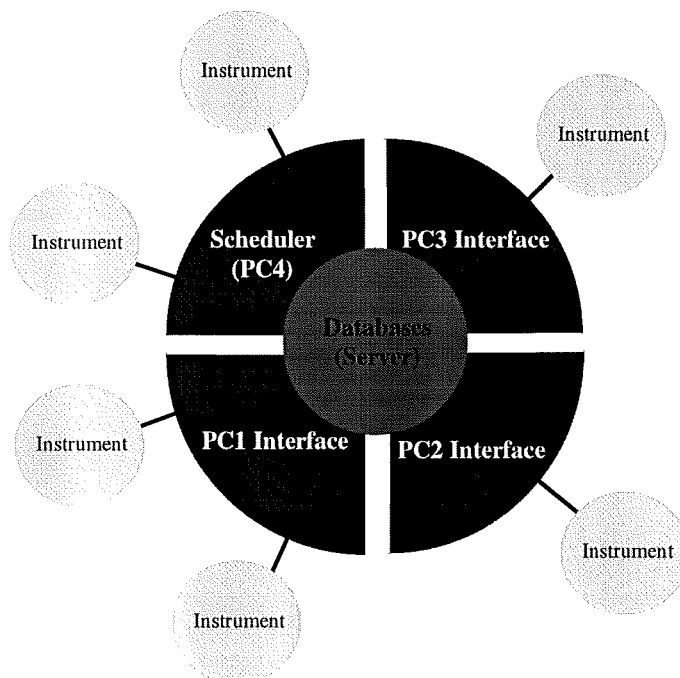


Figure 44: Database Structure



#### 6.4.4. Run-time programs

Once a method has been developed with the GUI, the software calculates the resources required. After the samples and resources are in place, processes are scheduled in real time. Traditional pre-runtime scheduling was not applicable for the RPAL system because this is a non-repetitive and non-deterministic process. Our aim was to be able to analyse several samples at the same time, in parallel, using different analytical methods and different operational parameters in the workstations. Method development strategies can be applied, and each step optimised. Another advantage of such a system is that the use of on-line quantification steps (such as LSC) will allow the development of an expert system in the future. At each step, the results will make the decision for the next step, via a decision tree developed by experienced analysts. Optimisation of processes has been reported (Wieling, 1994) but on a limited scale. Software device drivers were implemented to control and communicate with each RS232 interfaced instrument. When a station completes the task, the driver sets a call for the robot. A dynamic scheduler selects the next pick and place robot operation from all those calls, based on a set of priority rules. Once the sample is placed in the next station and the robot is back into a safe position, the scheduler sets a signal for the driver which downloads the operational parameters and starts the instrument.



**Figure 45: Interaction of run-time programs and instrument software drivers**

#### 6.4.5. System Responses to Errors

##### 6.4.5.1. Design-time Error Behaviour

Lewis and Norman, 1986, identified six possible ways in which a system can respond to its operators' errors. The actual examples are taken from human-computer interactions, but their underlying principles are applicable to a wide range of systems including this one.

###### Gagging:

A 'gag' is a forcing function that prevents the users from expressing unrealisable intentions. In a human-computer interaction, this could take the form of locking the keyboard to prevent further typing until the terminal has been reset. J.A.Raskin, cited by Reason 1990, inserted such 'gags' within his tutorial language system FLOW. Similarly in our system, if a user attempts to key in a character that does not form a legal command, it is not accepted.

###### Warnings:

Whereas the gag presents a block to anything but appropriate responses from users, warnings simply inform them of potentially dangerous situations. The user is left to decide the correct course of action. This is sometimes dangerous to let novices take decisions that can lead to dangerous occurrences, this is the reason why two different access levels have been implemented on this system: a user level, and a super-user level. The user level will issue warnings that will not be critical to the good operation of the system but rather the method entered by the user and operational parameters.

###### Do nothing:

As the name implies, the system simply fails to respond to an illegal input. It quite literally does nothing, and the user is left with the task of sorting out what went wrong. Such a device is only helpful when adequate feedback information is available. This 'do nothing' method is used where the level of competence of the person operating the robotics system would be surpassed and where special or advanced knowledge of the machinery is required.

###### Self-correct:

Whereas 'do nothing' method is the simplest error preventing technique, self-correct devices can be extremely sophisticated. Here, once an error (usually a programming error) is detected, the system tries to guess some legal action that corresponds to the users' current intentions. Very little of this behaviour has been implemented into the robotics system. Self-correct methods can lead to interpretation and misinterpretations, which is precisely what we want to avoid here.

Let's talk about it:

Some systems respond to user errors by beginning a dialogue. A useful example is the software driver interfacing the PLC with the host computer. At the beginning of each run, when the driver is initialised, a set of status bits is read and interpreted. This set of status bits reflects the state of readiness of the system. If one or more of these bits is not in the right status, the user is informed and prompted to make a few checks before attempting again.

Teach me:

On detecting an unknown or inexact input, the system quizzes the user as to what it was he or she had in mind. In short, the system asks the user to teach it. This approach is used more for the operational parameters entry and storage rather than the pure detection of errors in the system. For example, at the beginning of each run, the user is required to enter a method the samples are required to follow via the GUI. This method implies the configuration of each workstation necessary to perform the method. Each configuration is stored in a database for subsequent use but if the parameters entered for a given configuration are not suitable for the user, he is required to teach the system new ones that will be entered in the same database ready for use for the next run.

In considering the human contribution to system disasters, it is important to distinguish between two kinds of errors: active errors, whose effects are felt almost immediately, and latent errors whose adverse consequences may lie dormant within the system for a long time, only becoming evident when they combine with other factors to breach the system's defences (Rasmussen & Pedersen, 1984). In general, active errors are associated with the performance of the 'front-line' operators of a complex system. Latent errors, on the other hand, are most likely to be spawned by those whose activities are removed in both time and space from the direct control interface: designers, high-level decision makers, managers and maintenance personnel.

Rather than being the main instigators of an accident, operators tend to be the inheritors of system defects created by poor design, incorrect installation, faulty maintenance and bad management decisions.

#### **6.4.5.2. System Run-time Errors**

The automated system installed at RPAL features three types of errors: “Status”, “System”, and “Station” errors. The “Status” error is flagged whenever a workstation is not in the “IDLE” state when it receives the command to work from the supervisory program. This is a safeguard against programming and reset errors.

The “System” errors report problems at the system or laboratory level such as:

- No Vacuum
- No Compressed Air
- Safety Surrounds Open
- Power Failure (System on UPS)
- Emergency Stop Activated
- Fire Alarm Activated

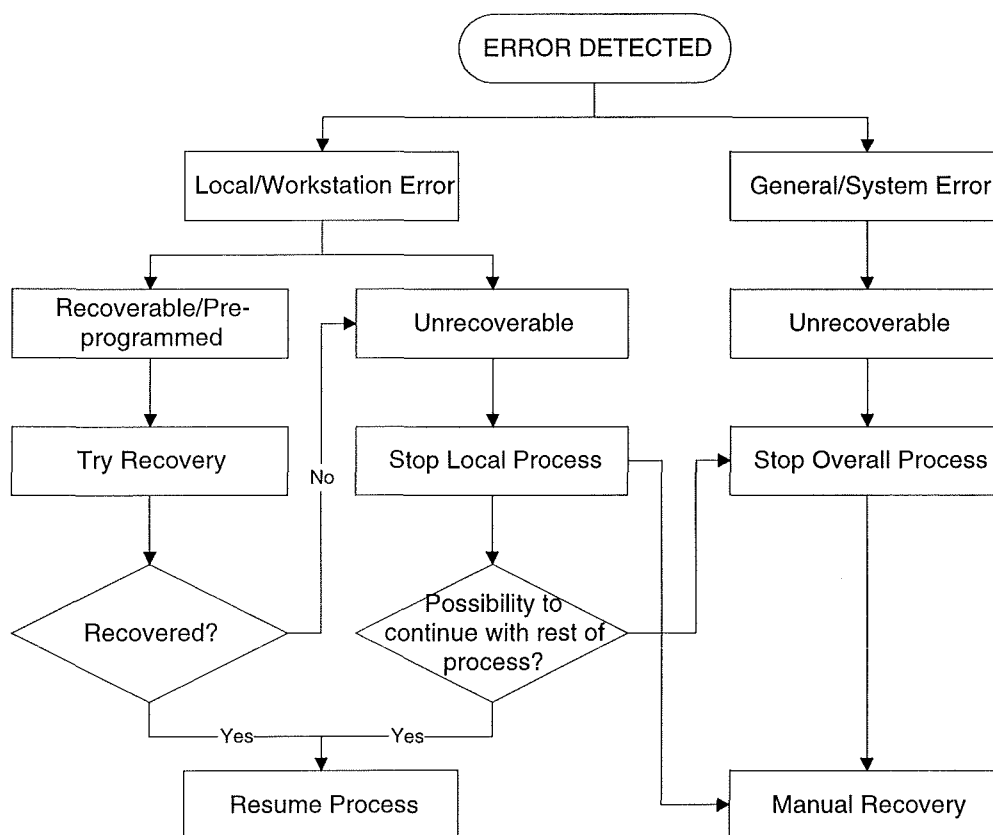
In most cases, this error type is unrecoverable and the overall process is stopped. The system must then be recovered manually and restarted.

The “Station” errors report problems at the workstation level such as:

- Pneumatic Cylinder Failure
- Lifting Mechanism Failure
- No Vessel Present
- Station Power Supply Failure
- Motors not Rotating
- Temperature not Rising

These errors are station specific and each workstation can have up to 20 of them.

This type of error does not always require the process to stop, only parts of it. Also, some may be programmed in so that an automatic recovery routine is implemented. This is the case of the filtering system where air is blown through a filter when the “filter blocked” error is flagged. See Figure 46.



**Figure 46: System Error Behaviour**

### 6.5. Decision Trees - Expert System

As the system evolves towards becoming an expert system, decision are gradually withdrawn from the operator hence, simplifying the analysis.

The current trial and error way of processing environmental samples, can in the future be programmed in based on the information gathered by the computer of a similar sample type processed in the passed. The idea is to identify what has worked and what hasn't worked for this type of sample and advise the user on the best or the recommended method to follow.

The implementation of decision trees in the future is based on the following strategy represented on Figure 47. This strategy was put together following a series of workshops involving the users of the system. Although a lot depends on a case by case basis, especially to compound and matrix, some general agreements were made:

- Extraction should be as simple and mild as possible to reduce/prevent degradation of compound and metabolite.
- Extraction should be as rapid as possible for the above reason, and to minimise equipment involvement.

- Solvent selection should be based on scientific criteria rather than 'experience' or gut feeling.
- Need to extract >90% of the Total Residue Radioactivity (TRR).
- If a re-extraction with the same solvent is <2% of TRR, then it is time to move on.
- Extraction solvents should be water soluble if the sample contains water, at least initially to ensure penetration of the matrix.

The sample is usually extracted once and counted on the LSC. If the radioactivity recovered is above 90% of TRR then the extraction is successful. Following a successful extraction, depending upon the level of radioactivity recovered, the sample is concentrated or not. If the activity is >10000 DPM, the sample can be aliquoted for High Performance Liquid Chromatography (HPLC). If the activity is less than 10000 DPM, the sample needs to be concentrated by evaporation down to a known volume, and then re-counted by LSC.

Some of the general features common to all users and analytical methods could be implemented right now, but a much thorough understanding of the results is needed in order to put a general decision tree control algorithm in place. Data must be gathered over a certain number of years in order to see any pattern emerging for some of the more complex decisions to take.

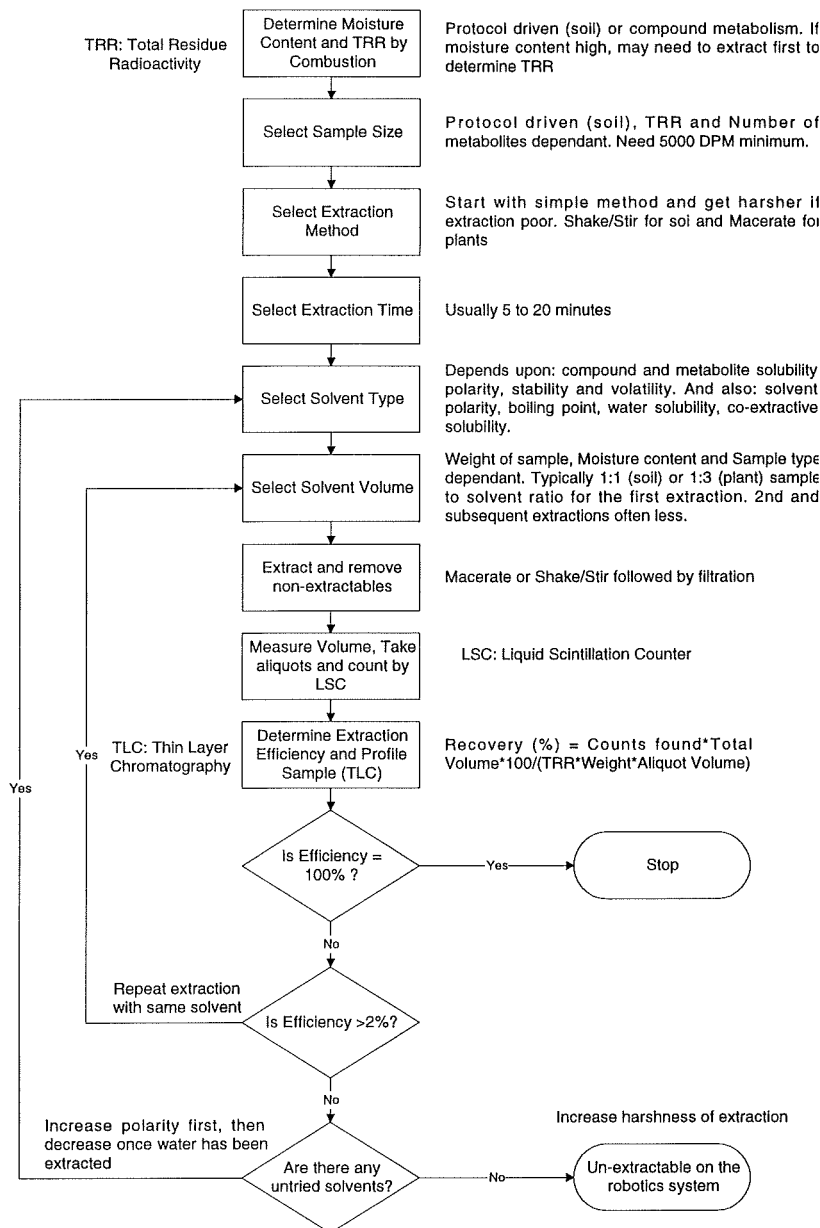
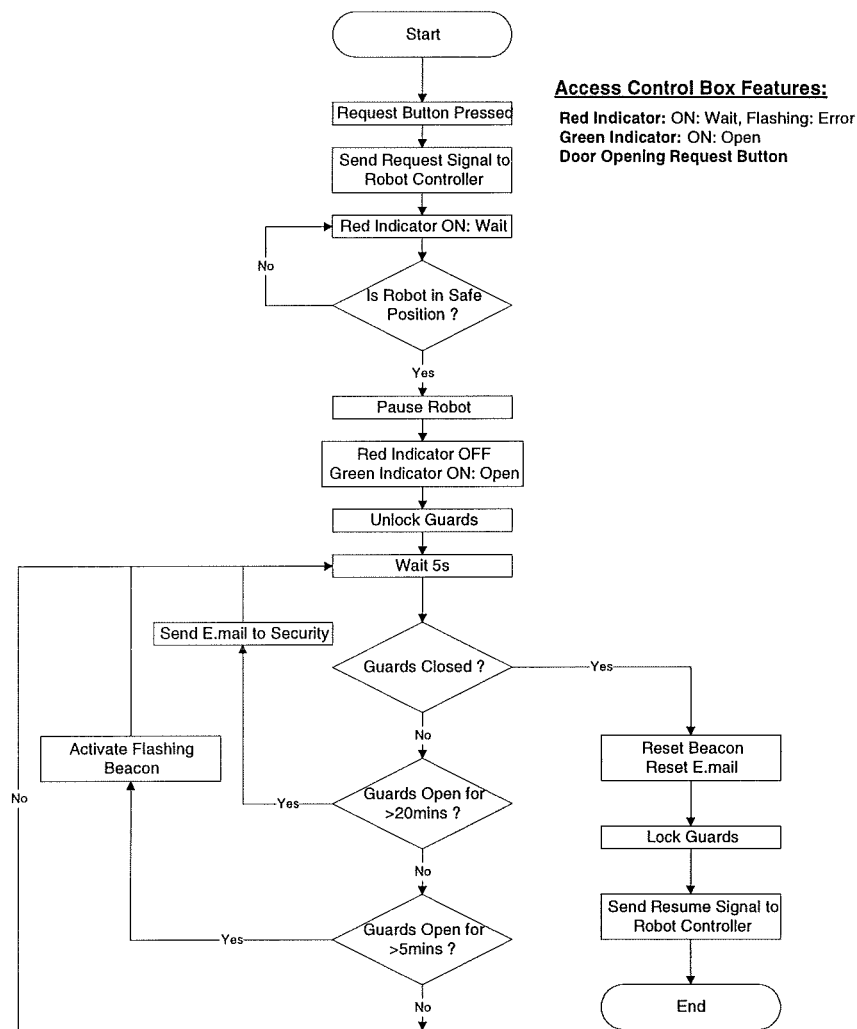


Figure 47: Sample Extraction Strategy

### 6.6. System Access Control and Emergency Stop

Based on the BS 5304, Safety of Machinery recommendations, an interlocking guard has been fitted around the automated system to prevent accidental access to moving parts. The robot will not move until the guard is closed. Similarly, the operator will need to wait until the robot is in a safe position to be granted access. The purpose of this system is to prevent personnel from loading or unloading samples and test tubes at the same time as the robot.

The controlling process for the guarding system goes as shown on Figure 48.



**Figure 48: Access Control Process Flow Chart**

The locking mechanism consists of a 24V solenoid attached to an extruded aluminium frame and a locking bracket attached to the window. The core of the solenoid in the de-energised position drops in the locking bracket preventing the window to be opened. As a feedback, every window is fitted with an inductive sensor that tells the system if it's open or closed. In the energised position, the core of the solenoid moves back inside the coil freeing the window.

The system has also been fitted with an emergency stop system featuring three stop buttons attached to the outside of the safeguards and three inside the guarded area. On pressing one of the emergency stop buttons, all moving parts are stopped, and pneumatic energy is released from the system. The robot is halted using one of its general purpose inputs. The emergency stop circuit fitted to the robot could be used in our case as the power is cut to the arm and the brakes are applied a fraction of a second later. This lag is significant enough for the arm to drop slightly under gravity. Tests have revealed that if the robot was carrying a test tube full of solvent, it would drop it on the track. It was

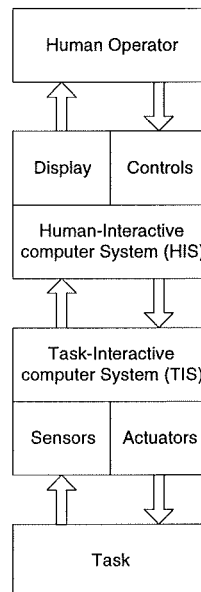


therefore needed to find an alternative to the robot built-in emergency stop. The robot is programmed to check one of its inputs continuously while in operation. In case of an emergency, this input is activated by one of the systems safety relays included in the system's daisy chain, see drawing in appendix.

## 7. DISCUSSION

### 7.1. Operator vs Designer Responsibilities

One of the most remarkable developments of recent years has been the extent to which operators have become increasingly remote from the processes that they nominally control. There is a real sense in which the computer rather than the human becomes the real actor, see Figure 49 (Moray, 1986). For most of the time the computer will be making the decisions about control, and about what to tell or ask the operator. The latter may either pre-empt or control or accept it when asked to do so by the computer. But normally, despite the fact that the human defines the goals for the computer, the latter is in control. This is the reason why adequate specification and analysis of the manual process is so important when designing an automated system where a computer is the heart of the system.



**Figure 49: The basic elements of supervisory control (after Moray, 1986)**

Lisanne Bainbridge (1987) of University College London has expressed in an elegant and concise form many of the difficulties that lie at the heart of the relationship between humans and machines in advanced technological installations. She calls them 'the ironies of automation'. Many systems designers view human operators as unreliable and inefficient and strive to supplant them with automated devices. There are two ironies here. The first is that the designers' errors, make a significant contribution to accidents and events. The second is that the same designer who seeks to eliminate human beings

still leaves the operator 'to do the tasks which the designer cannot think how to automate' (Bainbridge, 1987).

In an automated plant, operators are required to monitor that the automated system is functioning properly. But it is well known that even highly motivated operators cannot maintain effective vigilance for anything more than quite short periods; thus, they are demonstrably ill-suited to carry out this residual task of monitoring for rare, abnormal events. In order to aid them, designers need to provide automatic alarm signals. But who decides when automatic alarms have failed or been switched off? In an attempt to try and overcome these problems, Good Laboratory Practices (GLP) imposes that users and super-users of any equipment have a training record and that everyone must be trained and evaluated during some practical exercises before being allowed on the system. In order to help personnel operate the system correctly, a Standard Operating Procedure (SOP) is issued and approved by management. This SOP outlines step by step the succession of actions to take and checks to make to run the system safely. The SOP also points out the responsibilities of each person in an emergency or in case of an alarm thus removing any misunderstanding or misinterpretation. Hence, when an automatic alarm is generated, only a few persons are responsible to take particular actions and sign against them.

GLP is not particularly well suited to address these problems as it is not its primary objective. Some progress can still be expected though from the application of GLP rules on data integrity and traceability to our project. Some extrapolations need to be made as to the application of the rules to some areas of the system development. These assumptions can be helped by the Good Automated Laboratory Practice (GALP) document in use in the USA and more specific to automated & computer systems than the GLP UK set of rules.

## **7.2. Throughput and Performance**

The original intention of the automated system is to process approximately 70% of all samples entering the Environmental Sciences Department at RPAL. This represents about 30,000 samples a year which amounts to 5.8 samples an hour if the system operates 5 days a week, 43 weeks per year. This is an unrealistic target especially at the launch of a new system. The current throughput of the system is difficult to estimate as it is method dependant. If users decide to do only sample extraction on the automated system, then the throughput is only limited by the stirring time entered, and a 5.8

samples per hour throughput can be envisaged. If more work or full analysis is required on the system, then 5.8 samples an hour is an unrealistic target. Future use of the system will determine the primary use of the system. It has already become apparent that some of the workstations initially integrated into the system might be obsolete and might need to be replaced by more up-to-date ones or simply different ones. Hence, if the system is found to be used mostly for straight forward extraction-evaporation-aliquot-count routine work, some of the obsolete workstations may well be replaced by duplicates of existing ones.

The basis for the integration is the fundamental choice of the system layout. Is a multi-robot system or track mounted robot suitable, should we go for a centralised or distributed control system. All the answers to these questions are project dependant and are probably different in each case. The choice of having a single track mounted robot in our case is based on the fact that the various processes on each workstation are relatively long, leaving enough time for the robot to move test tubes around. If the processes had been much shorter, then the robot would have been identified as the main bottleneck in the system and another approach would have been taken. By optimising the automated cell layout using a computer model, it was easy to identify which step in the process would limit the throughput. It was found at first that the evaporation would be the most limiting step and it was cloned several times. The extraction workstations have now become the bottlenecks as every sample entering the system must be extracted first. System usage will dictate if there is a need for multiplying the extraction workstations. If radioactive samples are processed, then the LSC becomes a limiting step of the process. The LSC only processes one vial at the time and the typical processing time is 10 mins per vial. Actions have been taken so that the system gives priority to the bottleneck stations in the system i.e.: extraction and LSC, over the others and the robot will attend them in priority.

Some decisions regarding the control system have been compromised to keep the cost down. The choice of a centralised control system was made in the past but the system has gradually expanded and would now probably be more effective with a distributed control system, where each workstation would have its own controller. Some of the workstations developed specifically for this system are being considered for patenting purposes. If this aim is pursued in the near future, each station will probably gain at being equipped with its own individual controller.

### **7.3. Project Impact and Key Learning**

#### **7.3.1. At Company Level**

It is clear that this project is seen as a success as far as the collaboration between Middlesex University and Rhône-Poulenc Agriculture is concerned. The help provided by the University in terms of postgraduate and undergraduate student projects but also facilities are invaluable. There have been several cases during this project where several departments got drawn into the project, especially when mechanical, electronics and software expertise was required at the same time. A total of 14 student projects (both postgraduate and undergraduate), have been carried out or as part of the RPAL Automation Project. This project has seen the collaboration of three Teaching Company Associates over a period of 3 years: mechanical, software and electronics engineers.

A close collaboration between the different disciplines involved in the design and build of this project is crucial for a successful integration. Each person must understand what is required by the others and what the overall plan is. It is also very important that good communication occurs between the engineers and the scientists in the laboratory. This is often one of the most difficult tasks to achieve and requires sometimes a glossary of terms to improve understanding of each other.

#### **7.3.2. At Personal Level**

Being a Teaching Company Scheme, this project helped me develop some skills such as project and time management, team work, and project profitability through professional training modules attended during a two-year contract.

The nature of the research carried out during this project allowed me to present a paper at the 29<sup>th</sup> International Symposium on Robotics at Birmingham in 1998, alongside very experienced and highly recognised professionals in the field of advanced robotics, see paper in appendix. A second article on the RPAL automated system, for which I am a co-author, is currently being submitted for publication in the journal: *Laboratory Robotics and Automation*.

It also allowed me to register for an MPhil at Middlesex University for which this thesis is being submitted.

Lastly, this project opened out onto a permanent position at RPAL as Automated System Consultant.

## **8. CONCLUSIONS**

The system developed for RPAL integrates twenty-two workstations alongside a six-meter track mounted robot. The system is able to carry out a full analysis of soil or plant samples for environmental studies including sample preparation steps.

This system automates several labour intensive manual processes such as sample extraction, preparation and analysis leading to higher data integrity, higher throughput and better use of resources and disposables.

The overall system integration for which I was responsible occurred at two different levels: mechanical and electrical, at workstation and overall system level.

Each workstation was designed and integrated around system common features such as robot gripper and glassware from a mechanical perspective. The electrical integration was designed to allow effective control and communication between a five-computer control network and the workstations. The robot itself played a major role in the overall system integration. A CRS manipulator was chosen based on cost, technical specifications and reliability. The integration of the robot started from the gripper design, which allowed the manipulator to handle any vessel or calibration weight used in the system in a vertical orientation. The choice of a track mounted robot increases its reachable workspace considerably providing a larger work-cell with more workstations reducing the cost of a multiple manipulator/cell system.

As the main interface between in-house built or modified off-the-shelf workstations and the computer network, a PLC was used. Due to the diversity of communication protocols amongst all the workstations: some of them requiring either the PLC or a computer to operate others needing both. Commercially available instruments usually feature an RS232 communication interface, but very few of them are totally controllable through this link, and require either an additional manifold system for handling test tubes or improved hand-shake communication that can be achieved by the PLC, see Table 2. Many electronics and mechanical parts were designed and manufactured with the help of Middlesex University.

**Table 2: Workstation availability and integration work carried out**

Workstation (Supplier)	Integration work carried out
Balance (Mettler)	Rack designed and fitted.
Extraction (built in-house)	Electrical & software integration.
Evaporation (built in-house)	Electrical & software integration.
Centrifuge (Sigma with modifications by V.A.Howe).	Balancing workstation required.
Centrifuge balancing (built in-house)	Mechanical & electrical design and build.
LSC (Packard)	Additional feedback on vessel position and automatic start implemented.
Pipette (Gilson)	Additional start/stop status feedback implemented.
Vortex mixer (Heidolph)	Mechanical, electrical & software design and build.
Ultrasonic bath (built in-house)	Mechanical, electrical & software design and build.
Solvent Dispensing (Hook & Tucker)	Last drop collection system implemented.

Indistinct from the integration is the operational strategy of the system (Ogden, 1996).

The operational strategy of the system is based on various requirements:

- User requirements
- Process requirements
- Regulatory constraints

This resulted in a compliant and viable strategy for system operation to be implemented. This strategy is based on minimising user inputs and therefore errors while achieving a high level of integrity and flexibility. Depending upon the parameters entered and the method of analysis followed, the system will react in a different way in case of an emergency or an error. Error behaviours have been studied and recovery routines have been implemented to minimise risks of explosion and injury. Furthermore this system was designed to operate independently from the rest of the laboratory and therefore features its own power, gas and water supplies. In case of a power disruption the system is fitted with an UPS that will sustain the power for a limited time, enough for the shutdown routine to take control and safely stop the system. A close collaboration with RPAL site services staff allowed me to integrate the system in a laboratory environment, which necessitated some structural changes.

The automated system implemented for RPAL has the following advantages:

- It can be used as a method development tool where each sample can be analysed following a different method. Hence the users are allowed to devise their own method of analysis like in the laboratory while the practical work is always carried out in the same way by the robot.

- Each method/user configuration is stored in a database for subsequent use by a reporting tool. This also allows to gather information on the best methods used and the most successful ones for a given sample type.
- Once the system has been operating for some time, enough data should be available to implement decision trees and gradually withdraw decisions from the user. The best decisions regarding operational parameters for a given sample type will automatically be taken by the system that becomes the expert.
- Operation and parameters are simplified so that only basic computing skills are required from the user who simply drags and drops workstation boxes onto the drawing desktop of the GUI and link them together to construct the desired method.
- The final benefit of this system is to release analysts from repetitive and time consuming tasks to do more productive work.

The original intention of processing approximately 70% of all samples entering the Environmental Sciences Department at RPAL seems unrealistic especially at the launch of a new system. The current throughput is difficult to estimate as it is method dependant, but added requirements during the course of the project have led this system to become too diverse in its capabilities. Today's objectives have changed and a production system capable of higher throughput is becoming more important than a very flexible and versatile system. It has already become apparent that some of the workstations initially integrated into the system might be obsolete and might need to be replaced by more up-to-date ones or simply different ones. Hence, if the system is found to be used mostly for straightforward extraction-evaporation-aliquot-count routine work, some of the obsolete workstations may well be replaced by duplicates of existing ones.

Further developments of the system towards becoming an expert system have already started. Decision trees have been made to identify the logical reason why a given action is performed as opposed to any other. By retrieving the samples and process data from the database system, we should be able to identify which process is more effective for a given sample type and use it as the default process the next time the system is run. This work will involve some statistical analysis of the data and major reprogramming of the control system, but system's embedded intelligence will be significantly increased while reducing process or method design time.



Automated analysis will reduce the development time required for a new product and is thus considered strategic to the future business operation. It is hoped that the automated system will reduce the development time by up to one year and consequently enable new products to reach the market that much more quickly. The projected benefits for the Company in this project are, therefore, very significant.

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## 10. APPENDICES

### 10.1. Appendix A: Published Work

This paper was presented at the 29<sup>th</sup> International Symposium on Robotics (ISR'98), Birmingham, 1998. It is also available from the conference proceedings: pages 27 to 30. ISBN: 0 9524454 7 6.

### **Integration and Operation of a 6 DOF Manipulator For Automated Sample Analysis.**

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**Abstract - An insight into the integration and operation of a flexible robotic system is presented. This paper describes the integration of a sixteen workstation laboratory automation system based around a track mounted robot. The required level of operational flexibility of the overall system puts the emphasis on interfacing and controlling effectively a large number of instruments. The integration requires specific knowledge of serial communications, TTL and PLC technologies. Due to the vast amounts of data related to each workstation, a thin ethernet network is implemented to control the system. A network server is linked to several computers, themselves connected to serial devices including a PLC, the robot controller, and many workstations. A high level of flexibility and integration of off-the-shelf and custom devices requires specialist skills for both hardware and software. The development of this unique system is a breakthrough in the field of laboratory automation.**

#### 1. INTRODUCTION

The increasing flexibility of automation technology has enabled it to be implemented outside the traditional manufacturing environment, into industries where there is little or no repetition of tasks. The laboratory automation workcell at Rhône-Poulenc Agriculture Ltd (RPAL) performs a number of the more common techniques of analytical chemistry, and is configurable by chemists between runs [1].

The role played by this automated system is to process samples of plant or soil material through an analytical method leading to the study of the degradation of potential new pesticides in the environment.

Hardware integration and cell layout has been developed and optimised using CAD and Visual Interactive Simulation (VIS) software packages [2].

Due to the highly regulated nature of the business, this automated system is reducing human intervention to a minimum by using decision trees. Samples are moved between semi-autonomous workstations by a 6-axis track mounted manipulator, and decisions on

the value of the result and to whether or not re-process the samples are taken by the control system.

Finally, an overview of the control and operational strategy of the robot and the overall system is given.

## 2. SPECIFICATIONS/INDUSTRY REQUIREMENT

The automated system being developed at RPAL automates several highly manual analytical processes following one another in a constantly varying order. The succession of operations carried out on a given sample will only depend on the result obtained from the previous operation but also from the analyst undertaking the experiment.

The main objective of this system is to reduce lead time and increase throughput while putting the emphasis on the friendliness of reconfiguring the system between runs via a Graphical User Interface (GUI).

Being a tool for method development and optimisation, the system evolves gradually towards becoming an expert system.

From the information gathered during runs, a decision tree is implemented and responsibilities are gradually withdrawn from the user.

Data transfer, storage, archiving and retrieval are brought up to the Good Laboratory Practice (GLP) standards.

Cross-contamination, radio-labelled samples, and solvent compatibility are determining factors in the safety evaluation and validation processes.

Ultimately the automated sample analysis system will be interfaced to the global Laboratory Information Management System (LIMS).

## 3. HARDWARE INTEGRATION

### A. Manipulator and track

The chosen manipulator is a CRS A465 6DOF Manipulator with a similar kinematic representation to the KUKA 6/1 or PUMA 6-axis, see Fig. below.

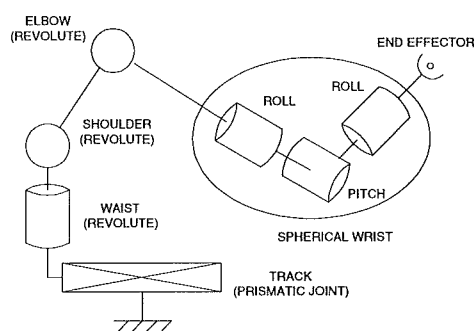


Fig.1: Kinematic representation of the 6 DOF CRS manipulator

For improved mobility the manipulator is mounted on a 6 metre track which allows it to have only one manipulator servicing all workstations instead of having several inter-linked robotic cells. The system is therefore more compact and easier to control but robot travelling and handling times must be kept to a minimum.

The necessity for the robot to hold test tubes in a vertical orientation at all times has put the emphasis on avoiding the 6 sets of singularities of this manipulator.

The CRS A465 is operated by a C-500 robot controller fitted with a SYStem Input/Output (SYSIO) module and a General Purpose Input/Output (GPIO) module. Those two modules allow the robot to receive and generate signals directly from and to

the PLC or other workstations without necessarily going through the computer directly linked to it. The SYSIO module allows greater integration of the emergency daisy chain of the robot into the overall system circuit.

### *B. Gripper design*

The design of the gripper is one of the most important tasks when integrating a manipulator. The gripper is designed so that it fits in all the current workstations, but future stations will have to be designed around the gripper. Therefore the design is robust and simple, and the number of robot re-orientations is kept to a minimum. A single hand rather than multiple hands is chosen to reduce lost time in hand changing mechanisms.

A servo gripper fitted with two angled fingers is used because its ability to control finger position makes it suitable to manipulate objects of varying size. A force sensor is integrated into the servo gripper so that real time feedback is obtained on the state of the fingers.

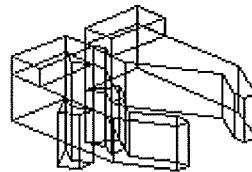


Fig. 2: Wire frame model of finger design

The design of the gripper shown above allows to grip a wide range of vessels and calibration weights with an outside diameter in the range: 11.5-65mm.

A 4-point grasping restraint is applied to any test tube for accessibility and implementation purposes.

### *C. Workstation design*

The kinematics of the CRS A465 allow it to reach in any workstation with almost any given position and orientation within the manipulator dextrous workspace which simplifies considerably workstation design. One of the main concerns when designing an automated system is to find the right balance between the amount of work carried out by the workstation and the manipulator. Due to the large number of stations, this system minimizes robot motions and handling tasks. Consequently the automation of some of the workstations was developed further allowing more autonomous operations freeing the manipulator from extra handling tasks.

Manual processes such as soil or plant extraction, sample transfer, and evaporation are sometimes difficult to mimic because they rely heavily on human intervention and inspection. Therefore other techniques such as optical level detection in evaporation, vacuum monitoring in filtration, and removing last drop from dispensing tip by solenoid actuation had to be found in order to automate these processes.

The system developed for RPAL had to integrate 16 workstations split into three categories: off-the-shelf, customized off-the-shelf and custom built equipment. None of the 16 workstations used in the cell could have been integrated directly without some modifications.

### *D. Cell layout*

In fixed based robot cells, reachable workspace is at a premium and geometric constraints often govern the location of workstations. The location of a robot within a workcell is often determined by reachability and mobility criteria; [3].

The more open arrangement of a track mounted robot system alleviates the shortage of accessible workspace inherent in fixed based robots and provides scope for future expansion.

A graphical simulation model of the robotic cell was generated using AT&T Istel's 'Witness' Visual Interactive Simulation software. Visual feedback from the animated display provides process insight and aids in finding more effective workstation arrangements within the cell, reducing cycle time and allowing increased throughput and capacity; [2].

From the simulation model, the robot was identified as the main bottleneck therefore effort has been put on reducing the transfer time to and from the frequently used workstations.

The multiplication of bottleneck workstations was also an obvious recommendation to increase the system throughput; [4]. See

Fig. .

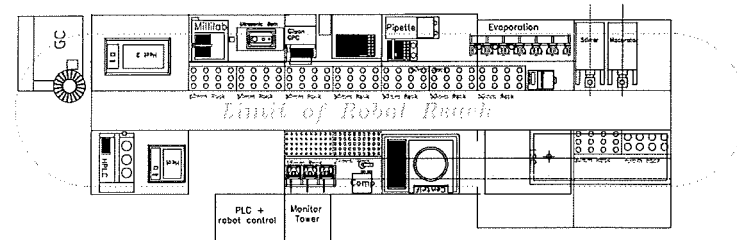


Fig. 3: Robotic cell layout

#### 4. CONTROL & OPERATION

##### A. Control system

The RPAL system uses five networked computers to distribute the workload of controlling twelve serial devices including the PLC and the robot controller, see Fig. . The network is also dedicated to handling the serial communications, controlling each workstation through software device drivers, executing overall supervisory control, carrying out robot scheduling, managing databases, displaying the Graphical User Interface (GUI) and generating reports; [5].

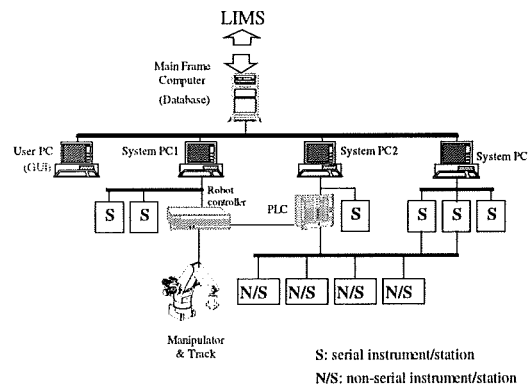


Fig. 4: Control Layout of Automated Sample Analysis



From the variety of stations integrated into this system, different types of controls are implemented. Commercially available instruments fitted with a serial interface such as RS232 or RS485 can be controlled directly via a computer. Non-serial instruments requiring analogue or digital signals are interfaced to the PLC. Some serial instruments used in workstations require an extra mechanism for handling glass vessels. Those types of workstations require both PLC and PC control, see Fig. .

Station control routines reside in their respective System PC. These routines will stand-by, monitoring the server databases until a switch field is set by the robot control application. This indicates that the vessel has been placed and the instrument is ready to work.

A real time robot scheduling approach is implemented here where workstations call the robot when they complete their task. A procedure will then select the next operation according to the current state of the system and a fixed set of priorities.

The degree of flexibility achieved here allows every sample to follow a different route while being processed alongside many others.

### B. Manipulator control & operation

The concept of safe positions is used to ensure that the robot moves down the track in a safe and predictable manner. These safe positions are with the robot arm and the gripper in a horizontal configuration, see Fig. . These safe positions enable the robot to travel between them without collision with equipment. Starting from the opposite end to the homing position the track has been divided into twelve 0.5 metre sections, numbered 1 to 12. The locations are called SR1 to SR12, S standing for Safe, see Fig. below. The variable %1 is used within programs to update the safe location. The track is then divided into East and West so that a location can be allocated to one side of the track or another.

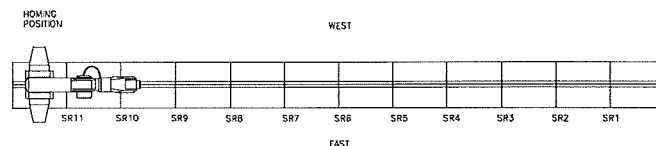


Fig. 5: Safe positions on robot track

#### 1) Rack Locations

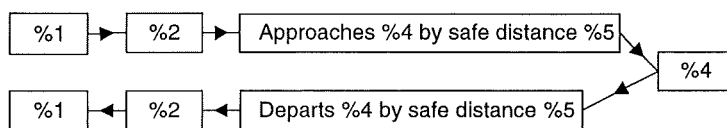
Storage racks for vessels are approximately 500 x 250 mm in size and contain a number of vessels arranged in a grid.

Each storage rack is associated with a vessel type, and a number of rack positions within the rack area, such that the locations are stored in an array (with R standing for rack). R5E50[001] is therefore rack 5, east, for 50 mm vessels, position number 1.

#### 2) Picking and Placing Vessels

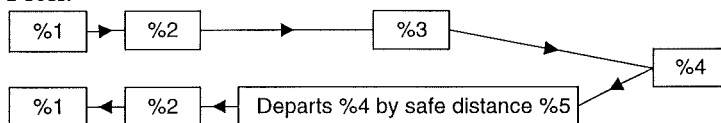
In order to pick up a vessel the robot (starting from the safe position defined above) must negotiate a safe route to the vessel in question, pick it up and return to the safe position by the same route. Similarly when placing a vessel the robot must negotiate a safe route between the safe position and the destination. In order to do this several intermediate points on its route to the final destination are defined. The variable parameters %2 (intermediate position), and %5 (safe distance above the destination) are used together with %4 for the final destination. Mixing these variables up between routines is liable to be disastrous and so the positions must be thoroughly checked

before running the routine. A test routine using slower speeds is used to test the procedure before running it at full speed. The routine thus goes in sequence:

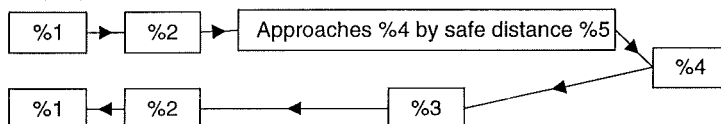


In order to pick a vessel from a terraced rack, approach from the side, and departure vertically is required, and the reverse to place a vessel. This variable is defined as %3.

Pick:



Place:



### 3) Gripping lipped vessels

In the case of a lipped vessel, the robot goes under the lip and closes the gripper with a force so that the force sensor detects the presence of the vessel. The gripper then relaxes so that when it departs vertically by 10 mm, the vessel slides down the gripper, the lip resting on the top of the gripper. A final force is then applied in order to securely grip the vessel.

### C. System operation

The first step to operate the system is to design a method using the GUI. A simple drag and drop operation is required to add workstations to the method. Each workstation must be linked to the next and configured as required in order to complete the process. At design time, the GUI ensures that two consecutive workstations are compatible with each other by selecting automatically the correct vessel size for the operation. From the method input by the operator, the system calculates the resources needed and issues a mapping for loading the samples on the racks.

The process will then start once an instrument calibration routine has taken place.

When a workstation is selected for operation by the scheduler, the pick and place parameters are downloaded to the robot controller with the command to move. Workstation running parameters entered by the user on the GUI are transferred to the selected instrument device driver via DDE (Dynamic Data Exchange) before the manipulator reaches it.

The workstation then awaits two start signals to begin its task.

The first start signal will come from the computer confirming that all parameters are downloaded successfully; the second signal will come directly from the robot controller once the robot has loaded the station and has reached the nearest safe position. Once the station has finished processing a sample, it sets a call in a given run-time database notifying the robot application that the unloading process can take place.

Workstations are prioritised by the robot scheduler according to their availability and throughput.

Each sample and its subsequent extracts are processed through the method entered by the user until it terminates on the rack where it was initially placed. If other subsequent

products of the original sample are to be collected from other racks, a mapping of the locations is issued to the user.

## 5. CONCLUSION

The choice of a track mounted robot increases its reachable workspace considerably providing a larger workcell with more workstations reducing the cost of a multiple manipulator/cell system. In choosing this approach, a computer simulation is indispensable to optimise robot handling times and workcell layout. Hence the emphasis must be put on robot handling operations when developing and integrating any new component into the system.

The integration of the robot starts from the gripper design which allows the manipulator to handle any vessel or calibration weight used in the system in a vertical position.

Robot path planning is also an important factor if the manipulator is not to spill any solvent on the track due to kinematic singularities.

The robot software application running under windows interacts with other applications via DDE. The robot application then communicates any command to the robot controller.

The control system is centralised and global due to the extensive workload. Local workstation control is reduced to a minimum. This strategy allows the system computers to communicate quicker and more effectively than with remote slower controllers.

The level of flexibility and friendliness of the automated sample analysis system at RPAL allows it to be used by anyone with little training.

The final benefit of this system is to release analysts from repetitive and time consuming tasks to do more productive work.

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## **A Robotic System for Sample Method Development using On-line Quantification and a Graphical User Interface.**

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**A robotic system has been developed, jointly between Rhône-Poulenc Agriculture Limited (RPAL) and Middlesex University, which automates method development, as well as being able to perform, simultaneous multiple-method, routine sample analysis. The system consists of twenty-one discrete workstations of varying complexity. Three different types were used: off-the-shelf ready to use, those requiring modifications, and purpose built. In order to achieve such a system, many of the discrete processes in the analytical laboratory have been automated in separate work-cells. Mechanical and electronic engineering is used to control and communicate with a distributed controlling computer system. A small industrial robot mounted on a six metre length of track, feeds the workstations as a pick and place manipulator. The control system gives the analyst full control of all the parameters associated with each workstation. For example, in the case of the centrifuge, acceleration, speed, temperature, time, and deceleration can be configured by the user. The system automatically opens the lid, loads the sample, balances the centrifuge, and after centrifugation indexes the rotor before removing the sample. A Graphical User Interface (GUI) allows analysts to use the system with minimal training, and to graphically represent the process in a familiar form.**

**Introduction** Analytical chemistry involves the extraction, clean-up and quantitation of small amounts of molecules from samples which are usually complex in nature. As a result the steps are labour intensive, and the quantitation step generally involves the use of sophisticated instrumentation. The use of pesticides requires methods for the analysis of the pesticide and their degradation products in the environment, for soils, crops and water. These require the study of the pesticides using soils and crops, which generally involves the use of radio-labelled molecules to aid following the degradation path. These studies are also labour intensive as analysis of the samples is required using similar techniques and quantitation methods. Combination of the study of degradation with developing a suitable method of analysis is beneficial in terms of efficiency of the process. Development of a fully automated system to achieve this objective has not been achieved before, and is reported in this paper.

**Automation** The benefits of automated analysis are potentially immense, but require a significant capital and resource investment. Many analytical techniques have been automated, and although transferring manual procedures to automated systems is, on paper, feasible, there are technical problems with automating an entire process. These technical problems need to be overcome in order to maintain flexibility of operation, and in the systems use, in the future, to justify the capital investment. These

problems revolve around the integration of equipment, not specifically designed to be integrated into systems. Extensive mechanical and electronic engineering is required in order to be able to control the equipment using software satisfactorily. In addition many workstations are not available and so require development.

Automated turn-key systems for the analysis of routine samples in the laboratory have been available since 1982, mainly utilising Zymark robots. Alternate robotic equipment systems, such as the HP ORCA, have also been used, but all suffer from disadvantages, including the need for specialist knowledge from the user. The use of systems integrators is the usual route for major projects, due to the specialist engineering, computing, and resources required to achieve a rapid development. However, as technology is rapidly changing, any future modifications cannot be easily done in house. The software code is unlikely to be available, and so a delivered turn-key system cannot be modified easily. A system with sufficient flexibility to be modified cannot be justified due to the high cost, and long development time to achieve it.

**Literature Review** The use of robotic systems has been reviewed by Majors<sup>26</sup>, Crook<sup>5,6</sup>. There are numerous references to the use of Zymark robotic equipment for the analysis of pesticide residues between 1985 and 1997, including Law and Jones<sup>16, 18-20</sup>, Owens<sup>30</sup>, Lemme<sup>24</sup>, Koskinen<sup>17</sup>. The only group to report the use of expert system software to control robotic systems is that of Isenhour<sup>2,10,12-15,21-23,36-38</sup> who between 1988 and 1994 describe the development of their system using the Zymark robot. Once a system reaches a certain size scheduling of tasks becomes important consideration. Corkan and Lindsey have discussed many of the issues<sup>4,25</sup> as has Murray<sup>28</sup>.

**Available Equipment** The advancement of computers, robotics and control systems has, in recent years, allowed rapid advances in laboratory automation. Stand-alone automated pipette and solid-phase workstations, as well as analytical quantification techniques are available commercially, from several manufacturers, and have over the last decade made significant impacts into the analytical laboratory. However, integration of stand-alone workstations into larger systems is difficult. This is because they have invariably not been designed to be integrated into larger robotic systems, and can be deficient in several areas. Most notably communications, robot access, and safety control are problem areas, requiring engineering knowledge. In addition the reliability of an integrated workstation needs to be excellent, as the integrated system reliability will need to be good, and the reliability of complex systems becomes less with increasing complexity. At minimum, they may require some modifications by the equipment manufacturer, and further in-house customisation, to enable some form of safety control in the automated system. Even if the equipment has a bi-directional RS232 interface, the protocol enabling communications may not be readily available. The available access area for samples may not be suitable for the robot, and some further customisation may be required to the equipment, or robot gripper, in order to access the sample area.

**Automated Analysis** Automation often requires the changing of traditional manual procedures so that they can be automated easier. Method development is usually done off-line using traditional manual procedures, and then transferred to the robotic system, which can result in problems with transferability between them. As a consequence the time taken to establish a new automated method is often considerable. Re-validation is then required, in the automated system, before routine analysis can commence. In addition, the sample preparation stage of an analytical method has always been a

limiting step in automation, due to the difficulties associated with its automation. Although this step has been automated, it was with limited control of the process. Thus the need to develop analytical methods, on robotic systems, is vital, especially if flexibility is required. These problems, and numerous others, have limited the effectiveness, so far, of automation in analytical chemistry applications.

Expert systems, using logical decision trees, enable method development to be automated, but requires the quantification of results in order to make the decision. Full integration of such systems has been limited and it is rare for an analytical method to have been fully optimised due to the considerable time taken to achieve such a situation. For example, optimisation, using automation of liquid-liquid extraction, has been reported. Although chromatographic method development software packages have been around for some time, it is only the final step in what can often be a long and time consuming process. Using a flexible system, all of these factors can be combined into a truly automated system. Once multiple samples are introduced into an automated system, scheduling becomes a problem, so a suitable way of scheduling the robot was required. The safety of analysts exposed to chemicals is also a consideration of increasing concern.

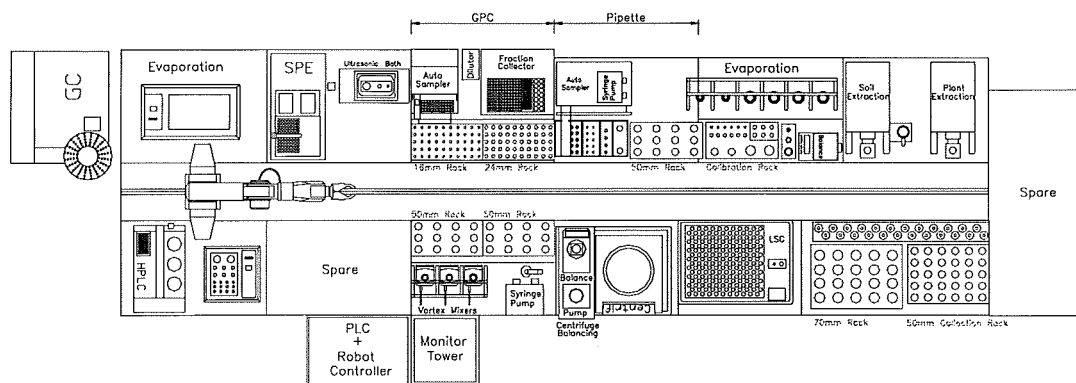
**Considerations** Robots suitable for laboratory applications have become more reliable and easier to incorporate into complex integrated systems. Some of the earlier problems associated with laboratory robotics are discussed by Shealey<sup>32</sup>. In order to provide a high degree of flexibility, built into an automated system, sophisticated programs are required. As the development of software is both expensive and time-consuming, turn-key systems are invariably rigid in their application. Writing the code in-house allows future modifications to be carried out and thus allows control without the need for re-negotiations with systems integrators. The optimisation of robot time between workstations using scheduling software is also an important consideration, as the running of complex systems is difficult to envisage. Although such software packages are available, they are usually tied to the systems integrator and come as part of a turn-key solution. Communications between engineers and analysts will invariably lead to misunderstandings, or at the very least insufficient dialogue resulting in incomplete understanding of the requirements. Specifications will not sufficiently detail the users' requirements since as automation evolves the user sees the benefits and requests modifications, thus changing the original specifications.

The availability of instrumentation with the capability to communicate (bi-directionally) with computer systems has advanced rapidly in recent years, with RS232 being the usual standard used. Manufacturers in many cases provide the software required to drive the instrumentation, and the necessary protocols to communicate with computers. However, there are several problems that are often encountered. First, even though different robot manipulators are available, moving a sample between workstations is often problematic, requiring a change in vessel, or workstation access is restricted. Second, the compatibility of stand-alone systems with other systems is invariably poor leading to software and hardware operating problems. Thus the integration of many stand-alone workstations into a total automation package is very difficult, requiring specialist expertise, and support from the manufacturer. The integration of robotics, with analytical equipment, including instrumentation also requires specialist engineering and computing skills. The use of companies specialising in this area is the traditional route to obtain such a system. Turn-key solutions provide the answer to many automation projects, but these invariably rely on the automation of set procedures, with

large numbers of samples. In order to automate a non-repetitive process each workstation needs to be configurable by the software in run time. The automation of non-repetitive processes is also impossible using traditional scheduling and control tools as workstations may be busy at the time required leading to delays.

**Project Management** Technology advances often make turn-key systems redundant before pay back times have been achieved. In order to reduce costs, minimise the risks, and be able to incorporate future requirements into the system, control of all aspects of the project, including the software code, was required in-house. In order to sell the system to users they were involved, at the outset, with the design of the system. A Total Quality Management (TQM) project was set-up to identify the major time consuming tasks performed in the laboratory, and those suitable for automation. As part of the project, off-the-shelf solutions for a number of tasks were identified and implemented immediately. These included data capture, temperature recording of sample storage areas, and the purchase of stand-alone automated workstations. The automated system described in this paper was the outcome of the remaining part of the TQM project. Initial ideas and concepts were discussed with automation integrating companies with regard to the feasibility of the concept, cost, and time to design and build. Although several companies were able to provide a solution, the risks associated with such a venture were deemed to be too high, such that future requirements could not be guaranteed without additional, unknown, costs. In order to be in control of the project, phase the development, and have the ability to axe the project at any time, it was decided to proceed, in-house. As Rhône-Poulenc did not have the necessary engineering and computing expertise, the final stage of the project was conducted in conjunction with Middlesex University. The University provided the expertise and technical support, and Rhone-Poulenc the funding. In addition the University was able to obtain government funding under the Teaching Company Scheme (TCS). This scheme is designed to introduce new graduates to industry, and train them to be effective, benefiting all parties. For this project three specialist engineers were recruited, each on a two year contract, and became part of a multi-discipline team with the analytical chemists at Rhône-Poulenc, and engineers at Middlesex University.

**Simulation of Process** In order to estimate the size of the system, the number of workstations required, and the performance of the integrated system to a range of different scenarios, a graphical simulation of manual processes in the laboratory was performed, using a discrete event simulation software package (Witness)<sup>33</sup>. This enabled an embryonic system to be developed prior to going into the expensive build stage. The workstations have been arranged in a logical sequence based on the results of the simulation exercises and integrated together in a robotic system. The purpose of the robot simply is to act as a pick and place manipulator, feeding the workstations with samples and vessels. The workstations are designated 'idle', 'busy', 'finished' or 'unavailable', and so using a simple system of workstation calls, the robot can be scheduled to move the vessels between workstations or racks. The simulation showed that a six metre length of track, with a 6 degree of freedom (DoF) robot would be required with twenty-one workstations arranged down both sides of the track. Several workstations required cloning in order to minimise bottle-necks. In particular the evaporation workstation was cloned to give six discrete evaporation units.

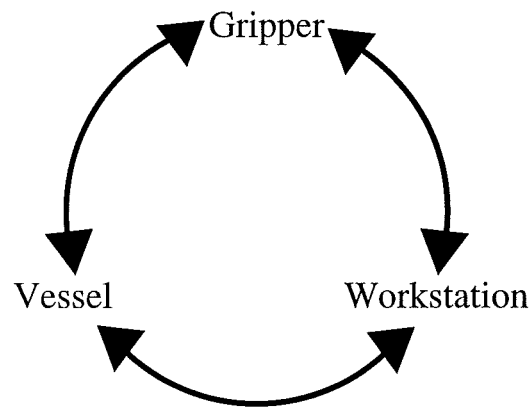


**Figure 1.** Layout of Cell showing positions of specific workstations

**Robot Selection** Robots designed specifically for laboratory applications have limitations over small general purpose industrial robots. The Zymark robot is a cylindrical robot with three degrees of freedom (DoF), and although suitable for many applications, it cannot easily be mounted on a track. The four DoF Hewlett-Packard ORCA robots promised much, with its superior software and control capabilities <sup>8,11,27,31</sup>. However, the lack of a waist, small pay-load (0.5 kg), and limitations on track length (2 m) again made it unsuitable for our application. Many industrial robots are designed for the heavy end of industrial applications, but the CRS small industrial robot combines all of the criteria that were required. It has a high degree of accuracy ( $\pm 0.05\text{mm}$ ), high pay-load (3 kg), 5 or 6 DoF, 6 m track length, and relatively low cost. It also comes with a teach pendant, and is easy to integrate into complex systems (programming, PLC, anthropomorphic configuration, software, and path movement). This robot has also been used successfully for other laboratory automation applications <sup>3,29</sup> most notable by North West Water. These factors were enough to satisfy us that this was the type of robot that we should use for our application <sup>9</sup>. The robot is controlled, using a generic program, to move to a particular location. This involves a set sequence of movements to guide it to retrieve or place a vessel, and then safely move away to a safe position. The concept of 'safe positions' allows the robot to move between any two safe positions, without fear of collision. Vessels have been specially designed to fit with the workstations and be easily moved by the robot. A uniquely designed gripper allows the robot to manipulate all sizes of vessels and to interact with any workstation.

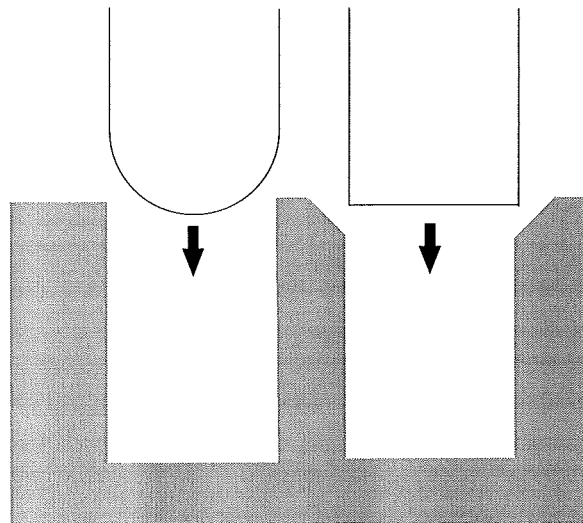
**Gripper Design** Although the robot came with a servo gripper, a pair of gripper fingers needed to be designed. The variety of vessels that were used in the manual processes was large, and incompatible with automation. In addition, access for some workstations, such as the centrifuge, was already pre-determined by the limited access available. Workstations that had to be developed would have to be designed around the gripper, and so they became constrained by the gripper configuration. The gripper design, the vessels used, and workstations thus became inter-connected, such that any change in one affected whether the other was acceptable. As a result the system design would need to be thought about early in the project in order to ensure that the gripper design was close to ideal.





**Figure 2.** Design Evolution of Robot Gripper.

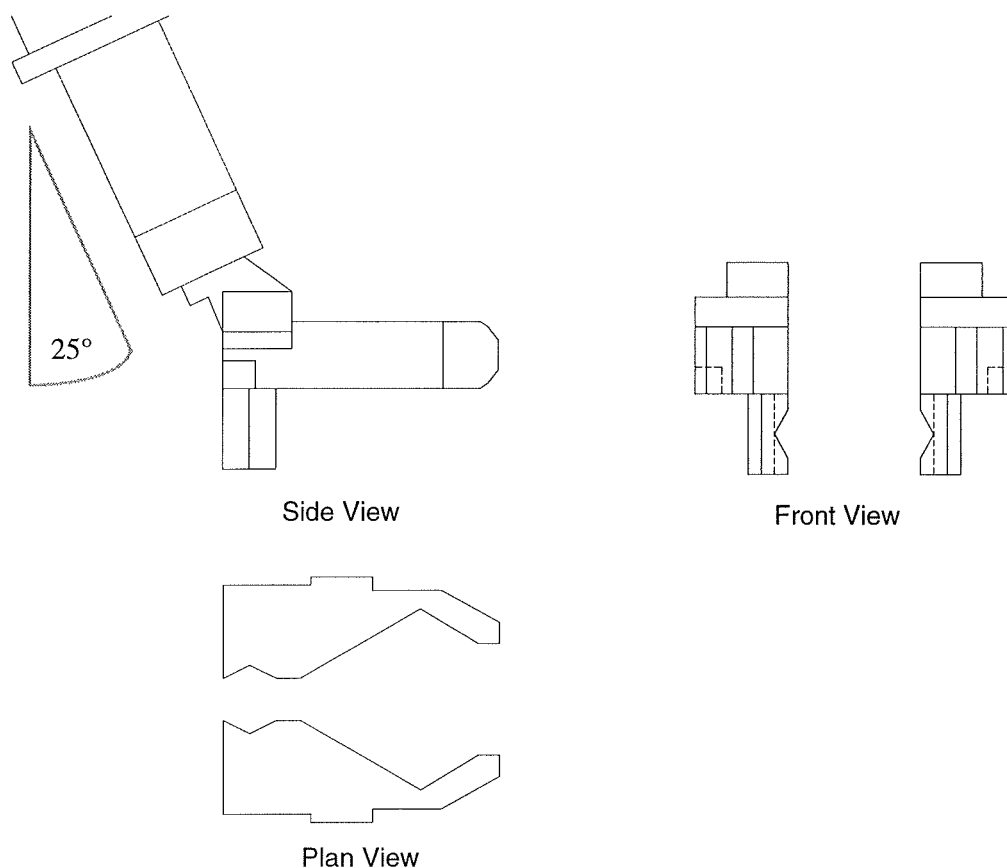
Optimisation of the shape and size of the vessels went a long way towards finalising of the gripper design, the final vessels being cylindrical in shape. This enables the robot gripper to grip the vessel, regardless of size in the same manner. The vessels were, wherever possible, selected with a round-bottom as accuracy became less of an issue. The round-bottom aids placing by guiding the vessel into the support rack during the placing operation, whereas a flat-bottom vessel needs to be accurately placed, and the rack location bevelled.



**Figure 3.** Round-bottom Vessel alignment

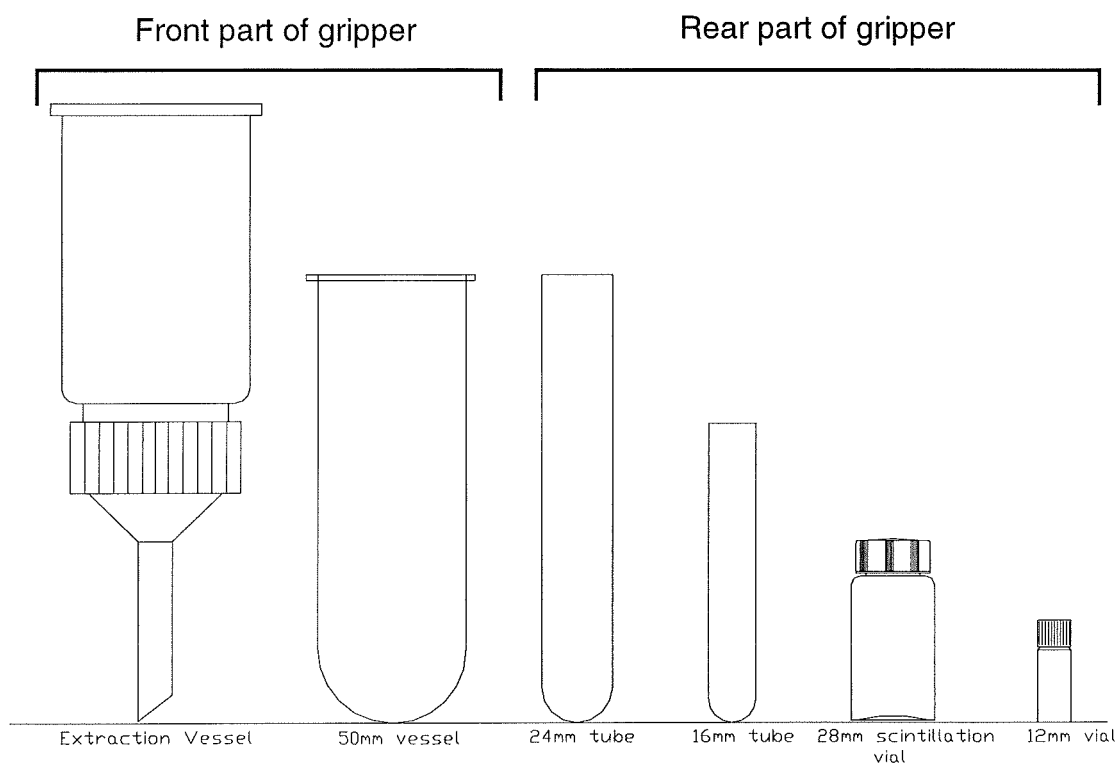
In addition, the larger, heavier, vessels were designed with a lip to aid robot lifting. The servo gripper, supplied with the robot, had a maximum opening distance of 50 mm, but the vessel diameters ranged from 12 to 70 mm. In order to overcome the problem of lifting the largest vessel, the gripper hand had to be open by a least 20 mm. This made it impossible to lift the smaller vessel, so two lifting positions were required on the same hand. Another solution was to use inter-changeable gripper hands, but the additional costs, and time delays, in switching hands during use did not look an attractive proposition, so considerable effort was put into the gripper design. Prototypes were made out of wood in order to obtain a satisfactory working gripper before the final design was machined in aluminium. The design evolved over a period, and was orientated at an angle of 25° from the vertical, in order to achieve the maximum vertical lift, which was required for the extraction workstation. In addition, rubber pads were

added to protect vessels from damage by the aluminium. The front part of the gripper handles the large vessels, the rear the remaining smaller vessels.



**Figure 4.** AutoCAD drawing of Robot Gripper.

**Vessel Selection** The vessels were optimised for the system, during the design stage, but the selection was, wherever possible, based on standard laboratory glassware. A total of six vessels were selected, based on the volumes of sample extracts used, and to optimise the transfer of aliquots. The extraction vessel is the initial vessel, containing the sample, and consists of a modified Schott Buchner funnel. The lower part is a polypropylene base funnel that holds a slotted polypropylene disc sandwiched between two solvent resistant seals, and secured with a screw-in Pyrex glass filter head. On top of the slotted disc is placed a suitable glass-fibre filter disc that is held in place with a stainless steel mesh. The soil or plant material is then added prior to running on the system. Three intermediate vessels are used in the system for the bulk of the sample work-up, and consist of carefully selected vessels enabling the minimum number of vessels, yet allowing the maximum flexibility of extract manipulation. The volume ratios between consecutive vessels are approximately five to one, allowing between 200 ml and 8 ml of solvent to be manipulated at the two thirds full mark. These tubes are 50 x 150mm, 24 x 150mm, and 16x100mm, the last two being standard test-tubes. Final extract vessels are the LSC vial, and a standard 12mm vial suitable for most automated chromatographic instruments (GC or HPLC). On-line GC and HPLC were incorporated into the design, and is the next phase of the project. The final vessels selected are shown in figure 4.



**Figure 5.** AutoCAD drawing of vessels used

**Workstations** In order to replicate the manual processes performed in the laboratory, and be able to automate them, modifications were required in the way that some processes were performed. It was felt that as long as the chemistry of the process was unaffected, the changes were deemed acceptable although, of course, validation of the modified process was performed. This resulted in a list of key workstations that would be required in the automated system. Once the type of workstation had been defined, the next stage was to approach commercial laboratory equipment suppliers in the hope that they would be able to supply suitable equipment. Unfortunately very few manufacturers were able to help, and so many workstations were unavailable. The list was then split into commercially available equipment, and equipment not available. This second list then required the design and fabrication of workstations around the user specifications. Due to the large number of workstations that fell into the latter category, these workstations were either designed, in-house by the project team, or as student projects at Middlesex University, either as final year degree projects, or as ERASMUS exchange student projects. These provided a number of interesting design solutions and prototypes, some of which formed the basis of final workstation designs. In particular, the evaporation workstation design, was a major break through for the system.

**Extraction workstation** The extraction workstation is usually the first workstation of any process, and as one was not available commercially it had to be designed. Wright<sup>35</sup> developed an extraction workstation, but it was not suitable for high volume use. Existing manual extraction procedures were numerous, and generally not suitable for automation. Physical agitation and filtering seemed to be the easiest method that could be automated, as long as control of the process could be achieved. The criteria for extraction, in combination with ease of automation, lead to the extraction workstation design being one in which soil was physically stirred, or plant material macerated, using over head devices, with solvent addition under semi-autonomous control. The type, number and proportions of solvent, extraction speed and time are user configurable in

the GUI. The extraction and collection vessels are loaded by the robot, the collection vessel is then raised to meet the extraction vessel, and both are raised to the stirring paddle or macerator head. Solvents are then added automatically, and a slight positive pressure is applied to prevent solvent dripping through. The process then starts, and after a set time vacuum is applied and the filtrate collected in the collection vessel. The vacuum is monitored to enable the end-point, or problems, such as blockage, to be detected. To achieve the necessary control a PLC program was written in which the valves, micro-switches, sensors, etc were controlled automatically.

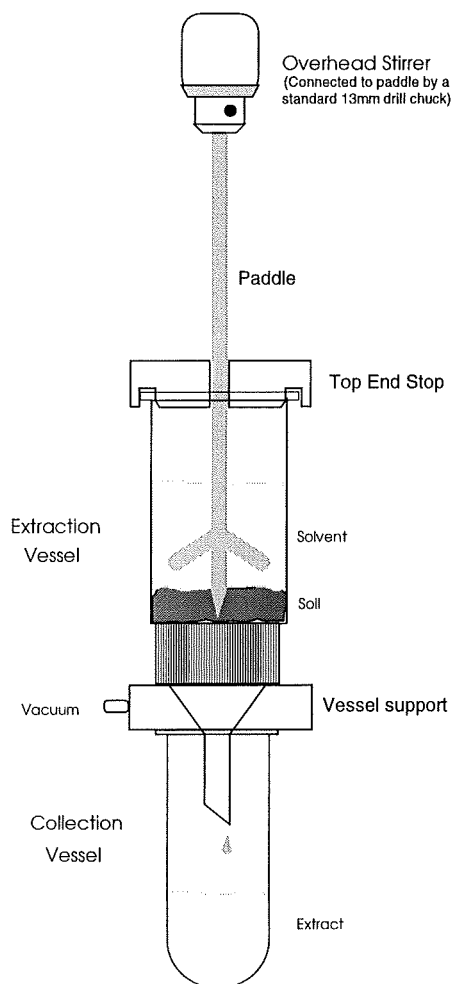


Figure 6. Soil Extraction Workstation Schematic

Workstation	Available (off-the-shelf) & Supplier	Degree of Hardware Customisation
Balance	Mettler	Minimal (rack only)
Extraction	No	Extensive
Evaporation	No	Extensive
Centrifuge	Sigma with modifications by V.A.Howe.	Minimal + Balancing workstation required.
Centrifuge balancing	No	Extensive

LSC	Packard	None
Pipette	Gilson	Minimal
Vortex mixer	Heidolph	Extensive
Ultrasonic bath	No	Extensive
Solvent Dispensing	Hook & Tucker	Minimal

**Software** In order to establish the hardware and software requirements a review of the different available options was performed in order to arrive at a control strategy<sup>7</sup>. The control hardware consists of a network of five computers, a Programmable Logic Controller (PLC) and the Robot Controller. The PLC, programmed in Ladder Logic, is used to control custom designed and built workstations using I/O control, and to interface them to the computer network. The pick and place robot routines were written in RAPL-II and stored in the robot controller. Drivers for serial equipment and overall control routines were implemented using Borland Delphi as the programming environment and Borland Paradox for the database. The software has evolved around the concepts of safety, flexibility of operation, and modularity for expansion.

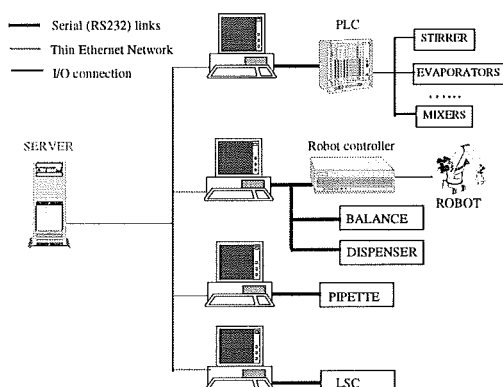


Figure 7. Communications Configuration

The system is managed through three main modules developed in-house: GUI, Database System and Run-time control Programs.

**GUI** Barnett described the principle of the user interface as long ago as 1988<sup>1</sup>. The off-line Graphical User Interface allows analysts, with minimal training, to develop a method via the computer screen. All workstations are represented graphically, giving the user complete flexibility for the analysis. Samples can be split into aliquots and processed with any of the analytical techniques that have been integrated into the system. Methods are built by dropping workstations in the desktop, configuring them, and joining them to previous stations. The GUI interacts with the user in order to guide the analyst through the method design process. The system checks sample volumes, vessel compatibility and workstation parameters at every step of the process. This ensures that only feasible methods are stored for running. The method development and system set-up capabilities of the interface also contain an integrated reporting tool that generates formatted reports containing all the data, after the method is run.

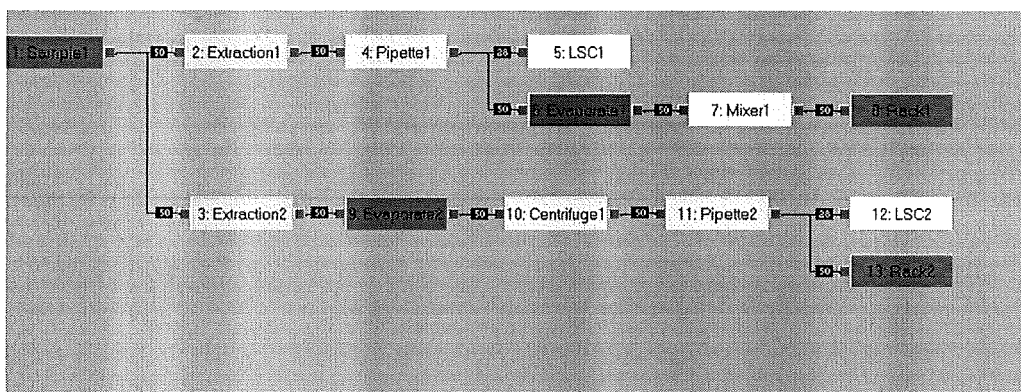


Figure 8. Method entered with GUI

**Database System** The Database system is more than a data storage tool because it has control purposes and maintains GLP. It consists of a net of databases distributed in three sets. The first one, the “User-time” set, is functional at the design stage. These permanent databases store all the information related to methods designed with the GUI, such as user, station parameters and connections. The temporary “Run-time” set is used by the control programs. When a method is run, its related information is transferred from the User-time databases to the run-time ones. Controlling computers use these databases firstly as a source of information to know which operational parameters to download each time and also, to synchronise and schedule instruments and robot operations. In addition, on-line generated data, in the form of weights, times, errors, etc. are stored here. Once the process is finished, all the relevant data from the run-time set is transferred to a permanent set of “post-run” databases. These are exclusively related to the particular run of that method and are used for reporting purposes.

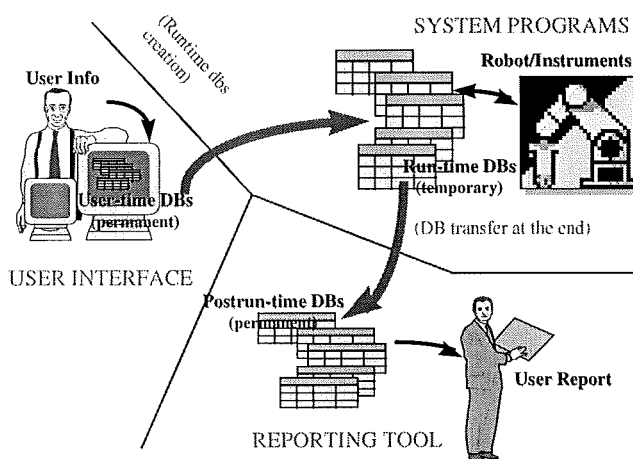


Figure 9. Database Structure

**Run-time programs** Once a method has been developed with the GUI, the software calculates the resources required. After the samples and resources are in place, processes are scheduled in real time. Traditional pre-runtime scheduling was not applicable for the RPAL system because this is a non-repetitive and non-deterministic process. Our aim was to be able to analyse several samples at the same time, in parallel, using different analytical methods and different operational parameters in the workstations. Method development strategies can be applied, and each step optimised. Another advantage of such a system is that the use of on-line quantification steps (such

as LSC) will allow the development of an expert system in the future. At each step, the results will make the decision for the next step, via a decision tree developed by experienced analysts. Optimisation of processes has been reported by Wieling<sup>34</sup> but on a limited scale. Software device drivers were implemented to control and communicate with each RS232 interfaced instrument. When a station completes the task, the driver sets a call for the robot. A dynamic scheduler selects the next pick and place robot operation from all those calls, based on a set of priority rules. Once the sample is placed in the next station and the robot is back into a safe position, the scheduler sets a signal for the driver which downloads the operational parameters and starts the instrument.

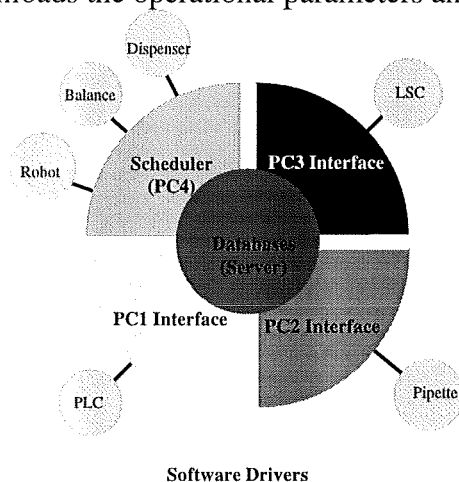


Figure 10. Interaction of run-time programs

## References

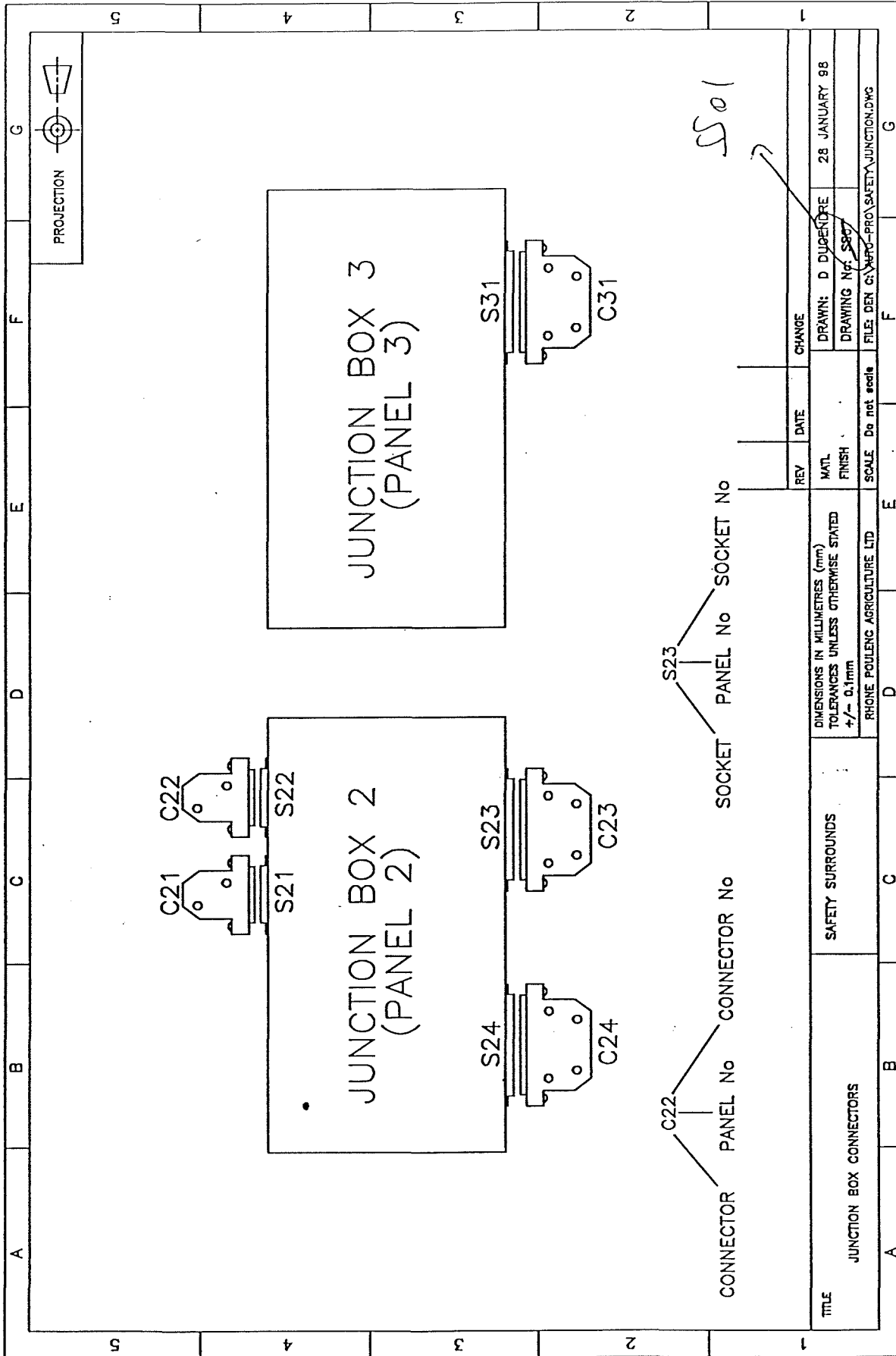
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## **10.2. Appendix B: Mechanical Drawings**

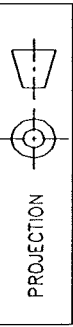
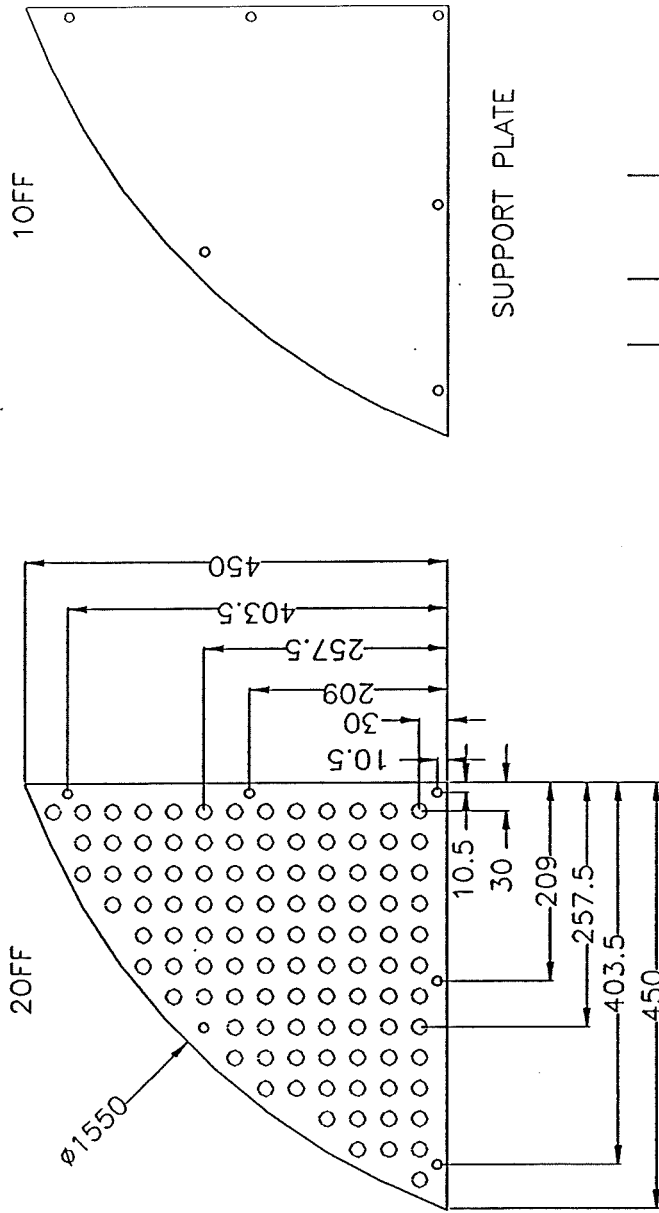
### 10.2. Appendix B: Mechanical Drawings



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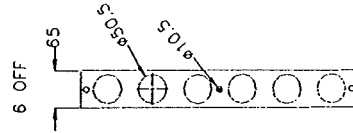
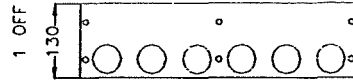
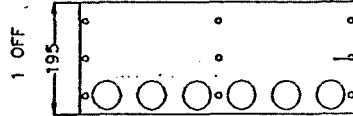
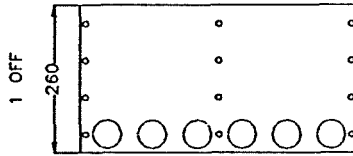
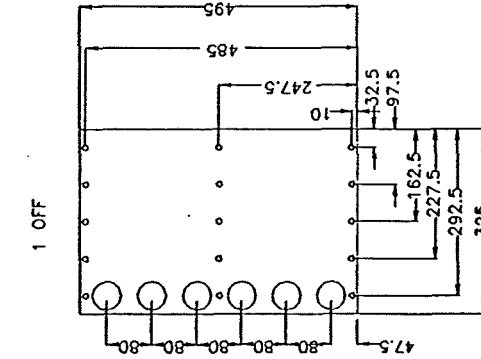
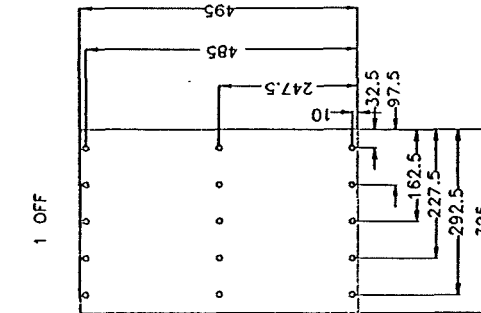
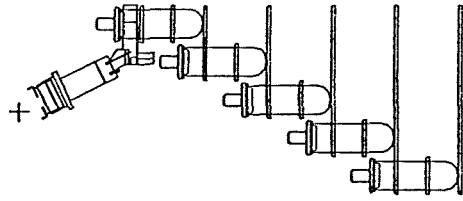
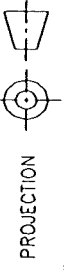
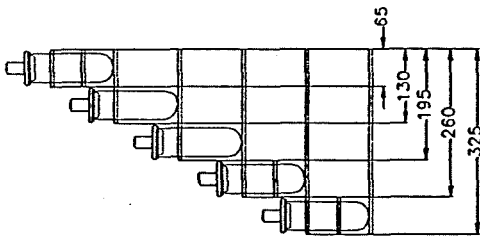
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All other tolerances: +/- 0.5mm



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DRG NO. RK 02

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 Fixing holes diam. 10.5 +/- 0.5mm  
 All other tolerances: +/- 0.5mm



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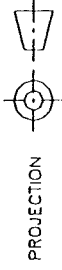
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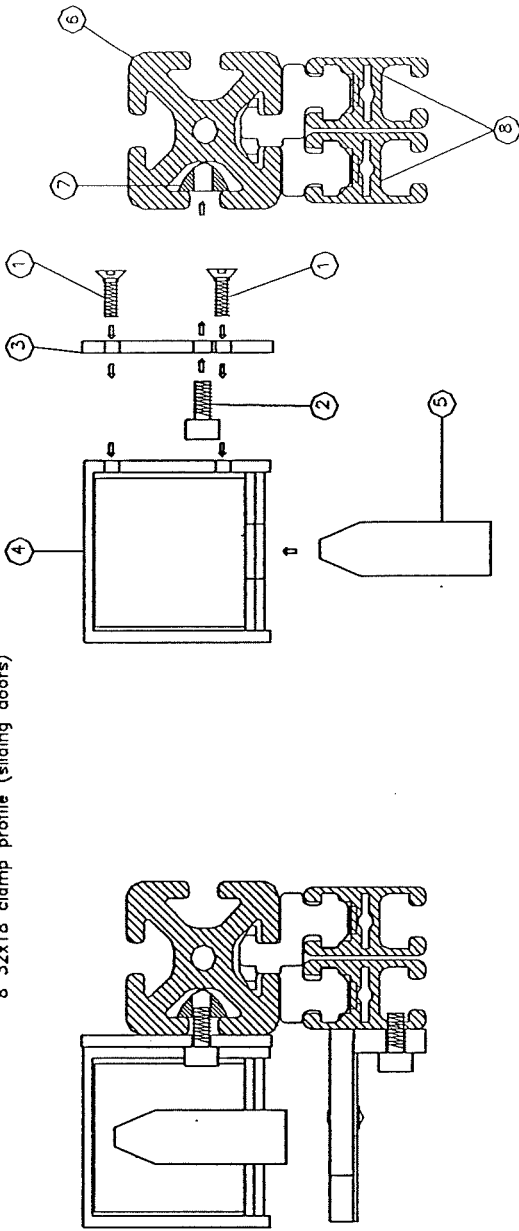
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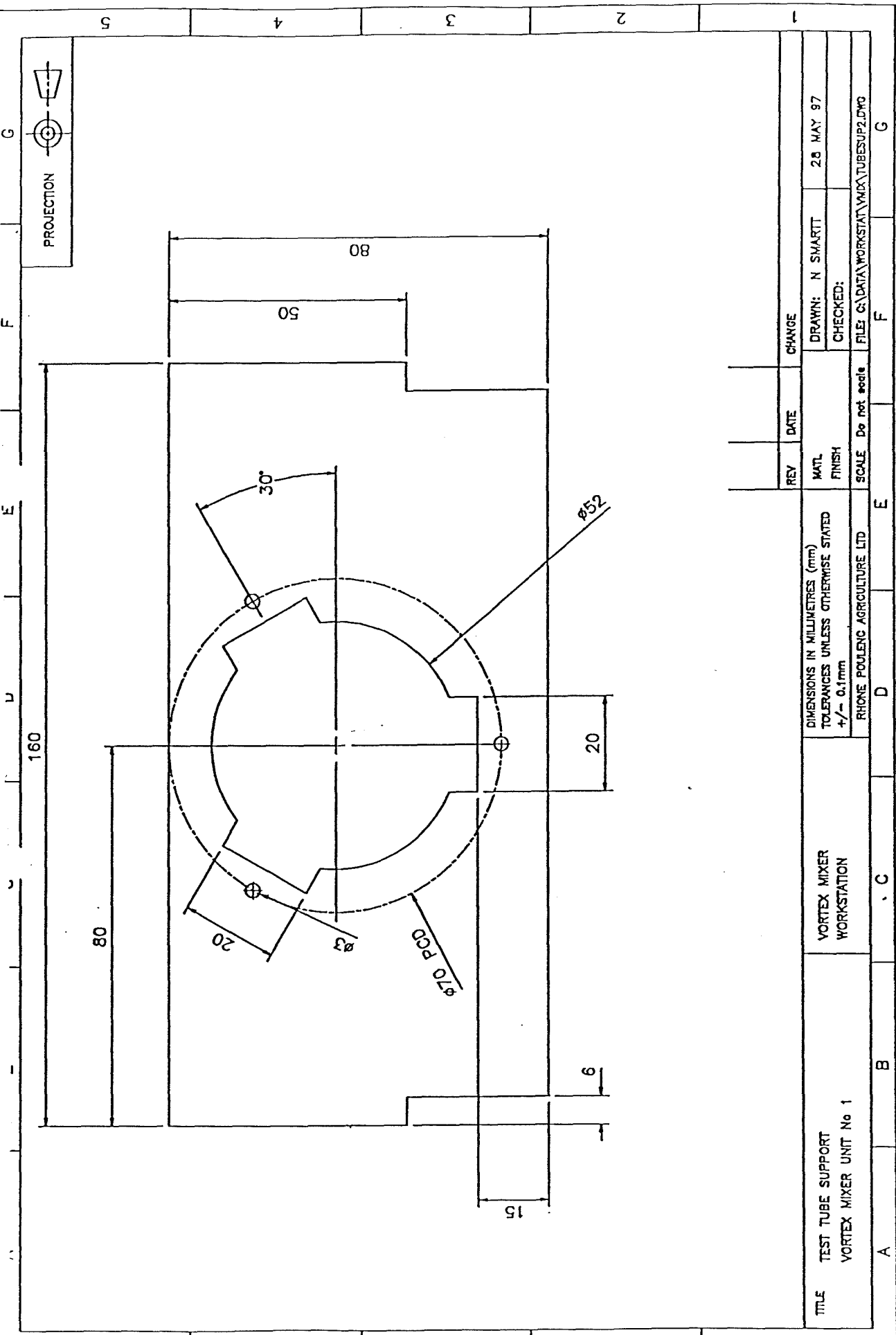
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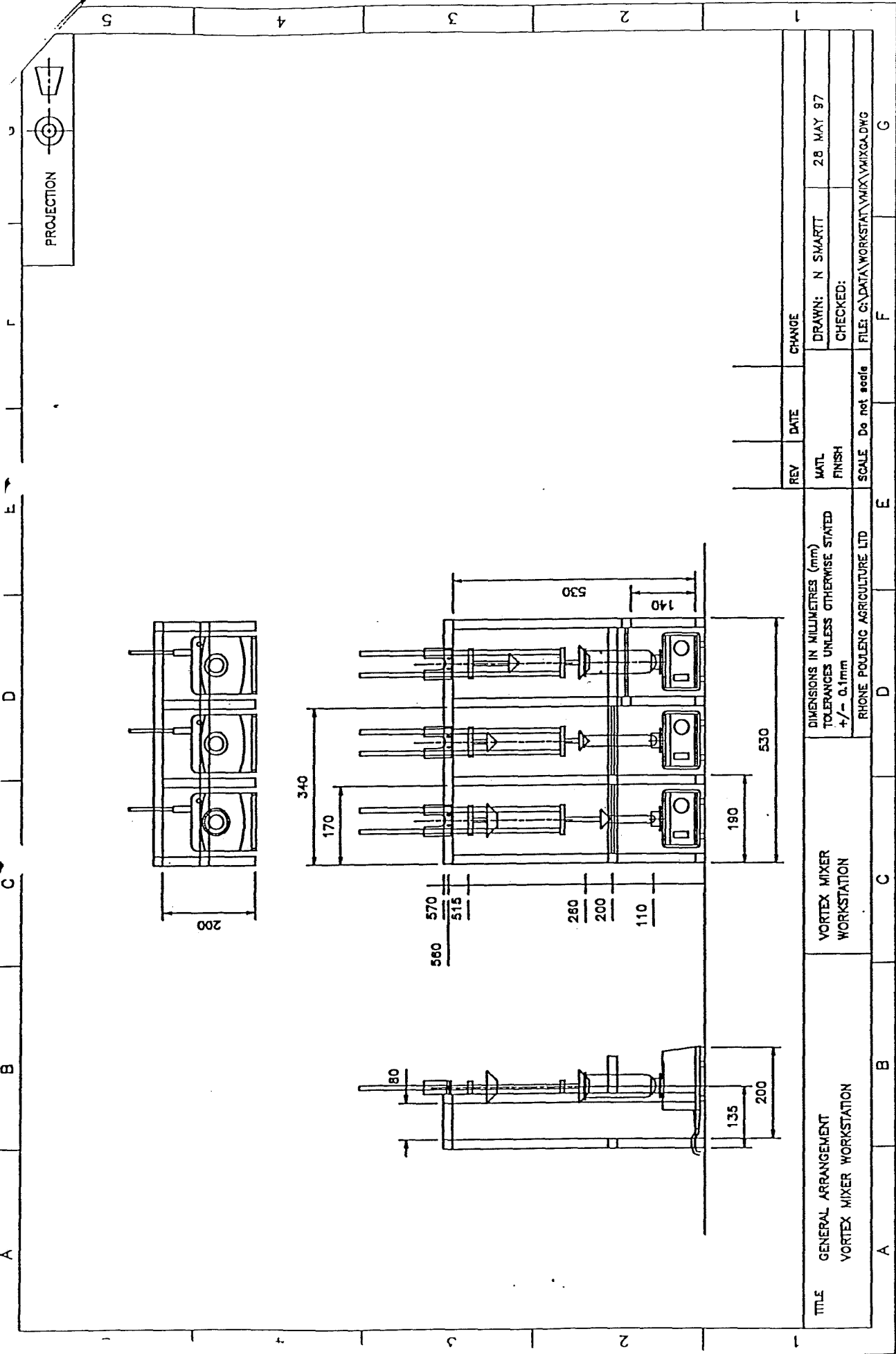
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- 2 M5x10 socket
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- 5 Plunger
- 6 40x40 aluminium profile (frame)
- 7 T-slot nut M5
- 8 32x18 clamp profile (sliding doors)



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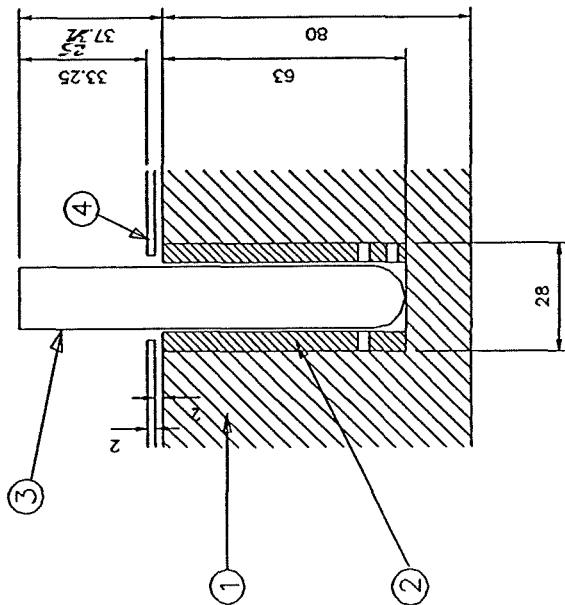


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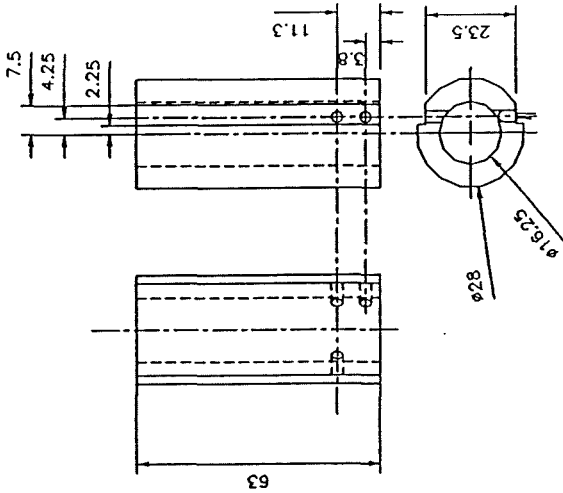
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PROJECTION



HEATING BLOCK ASSEMBLY

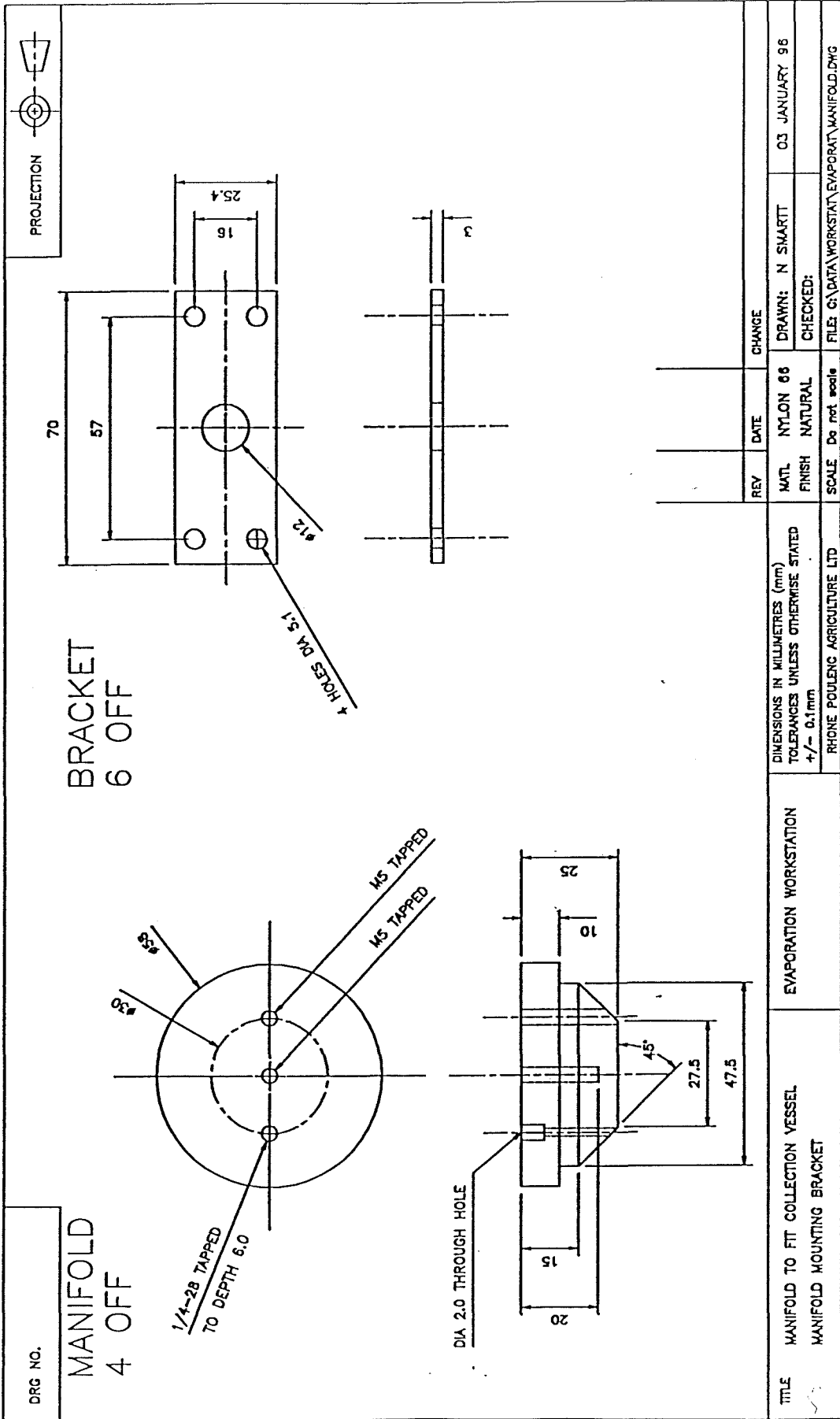
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- 2. INSERT
- 3. 16MM VESSEL
- 4. COVERING PLATE

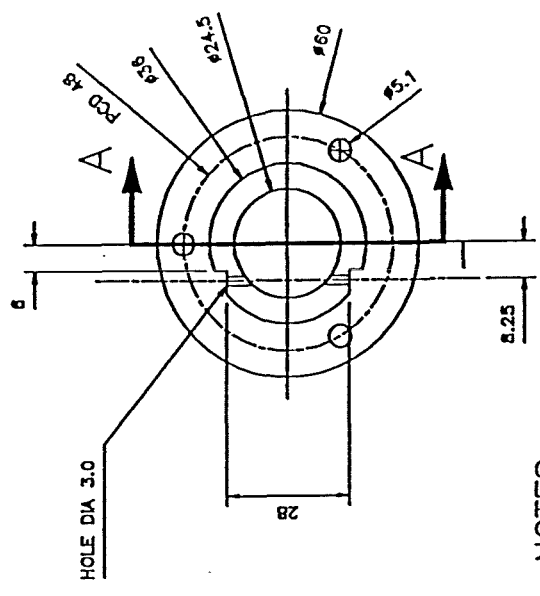
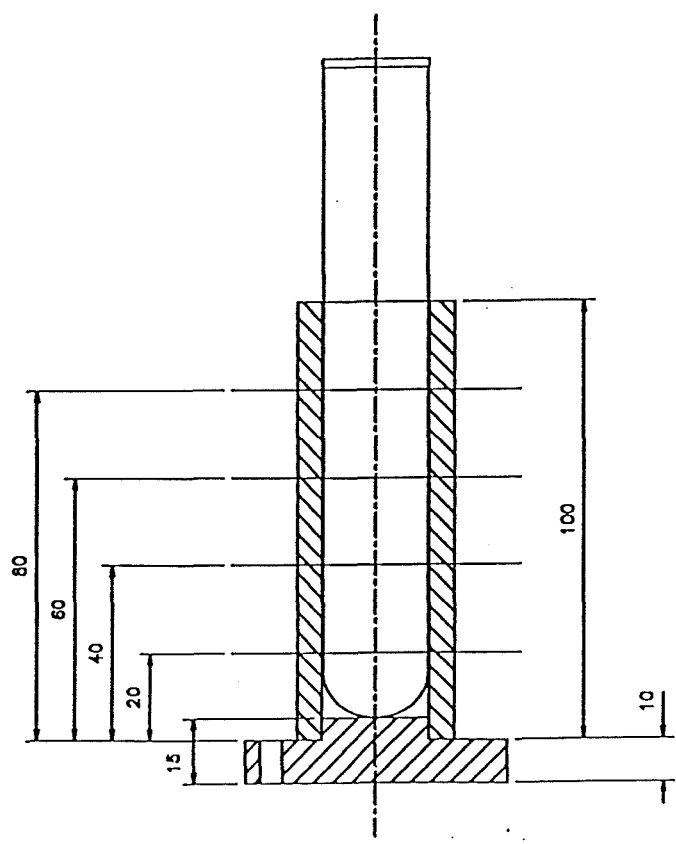
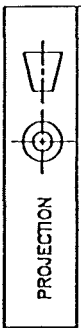


INSERT FOR 16MM TUBES

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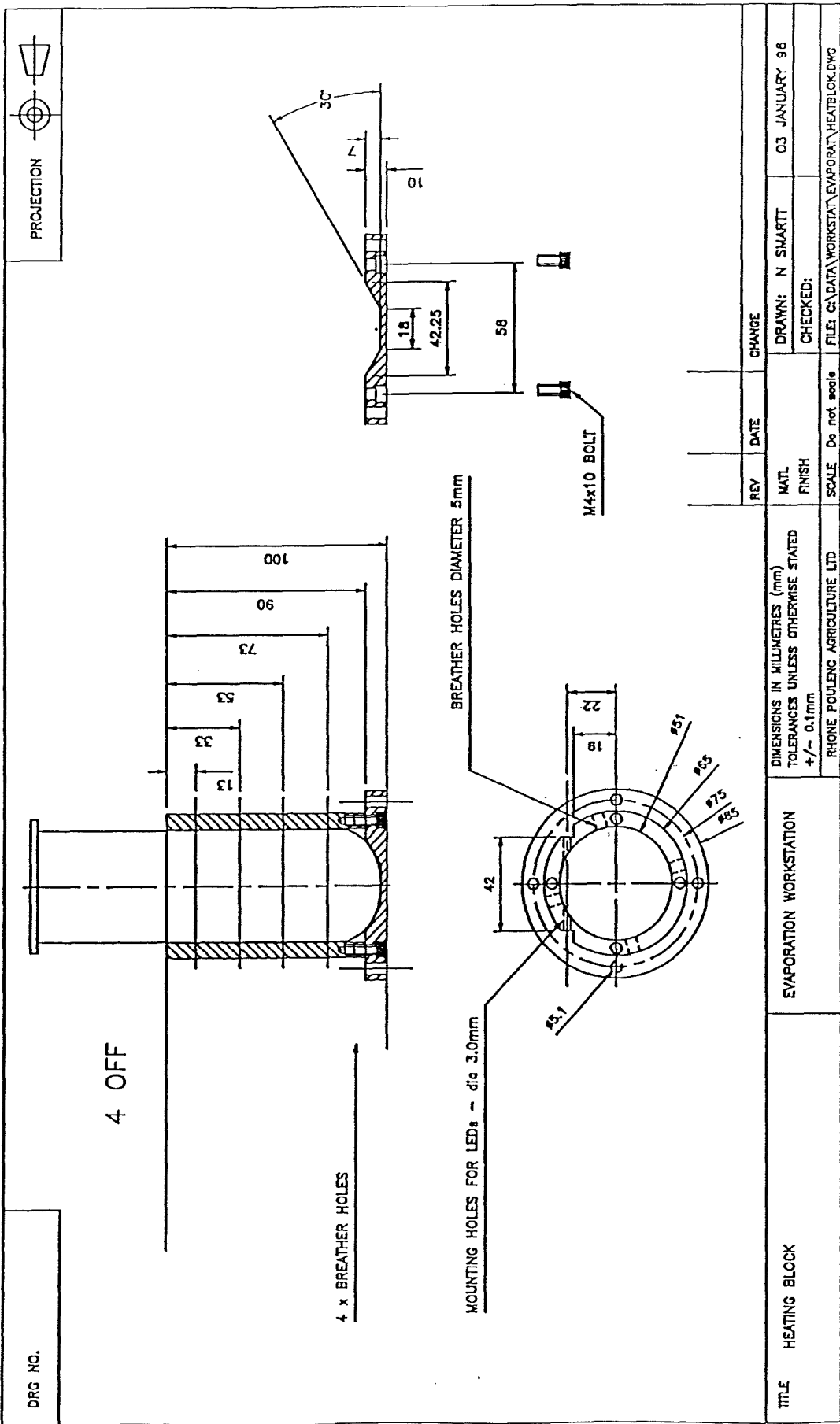


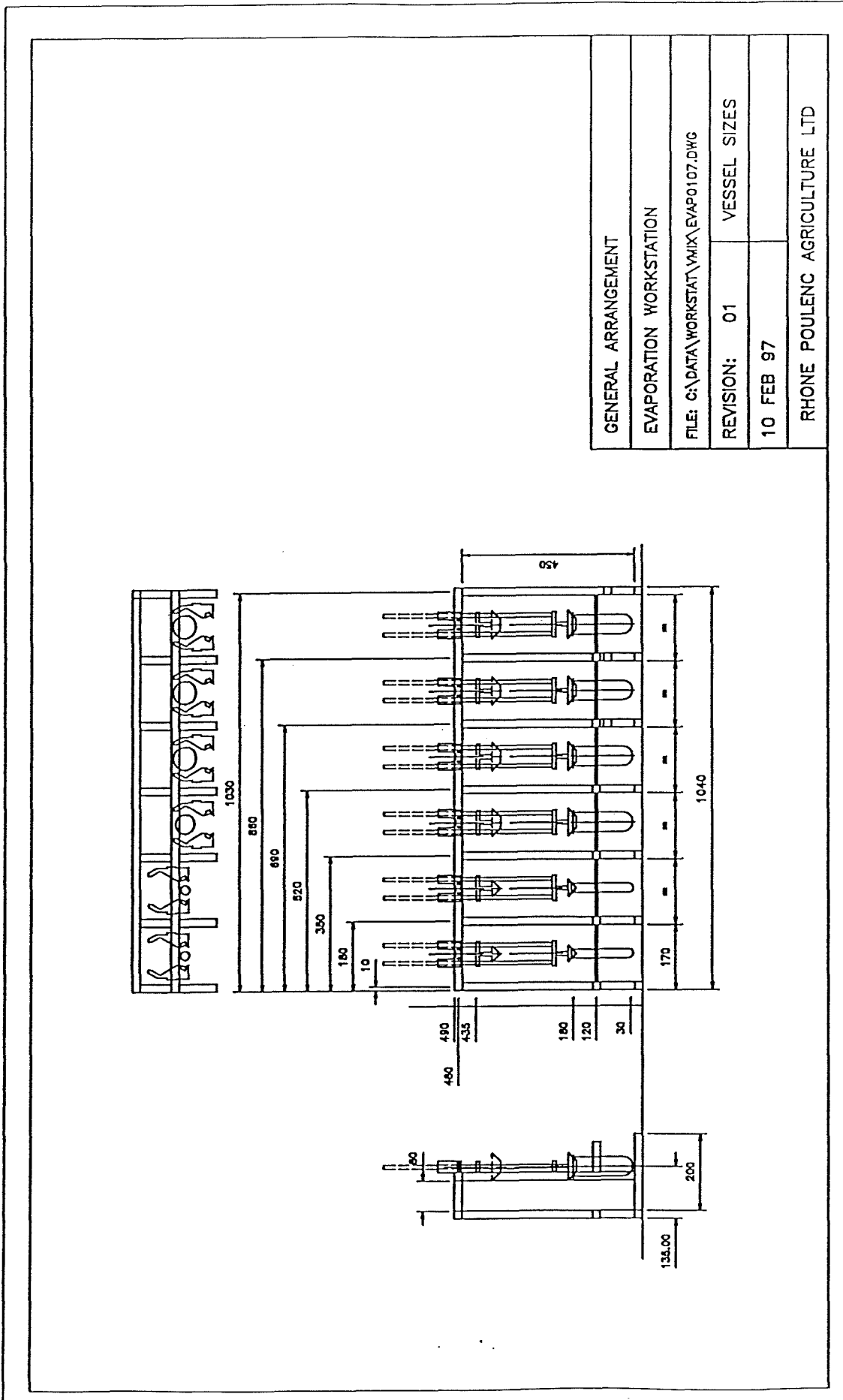




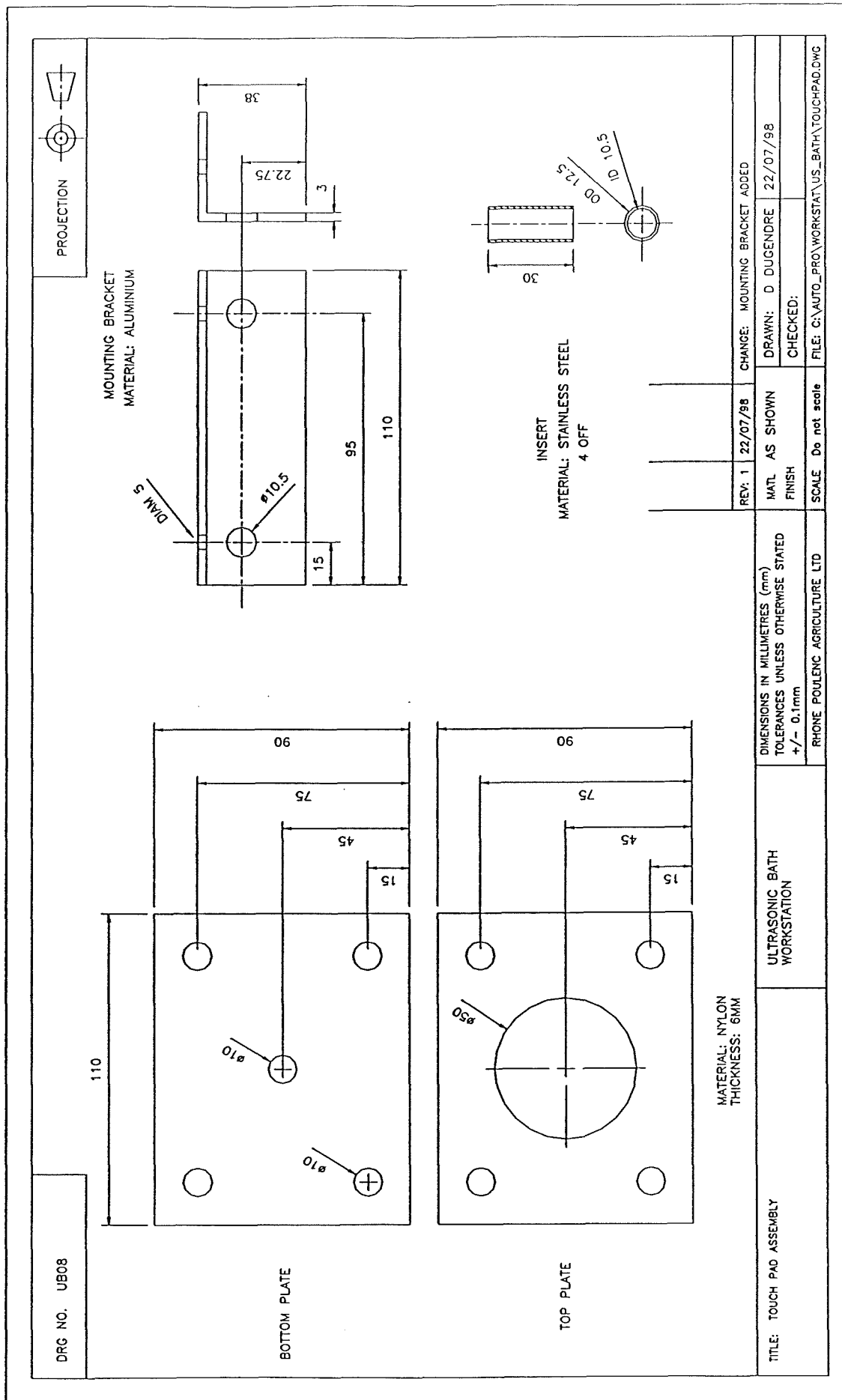
NOTES  
DRILL HOLES FOR IR SENSORS FIRST

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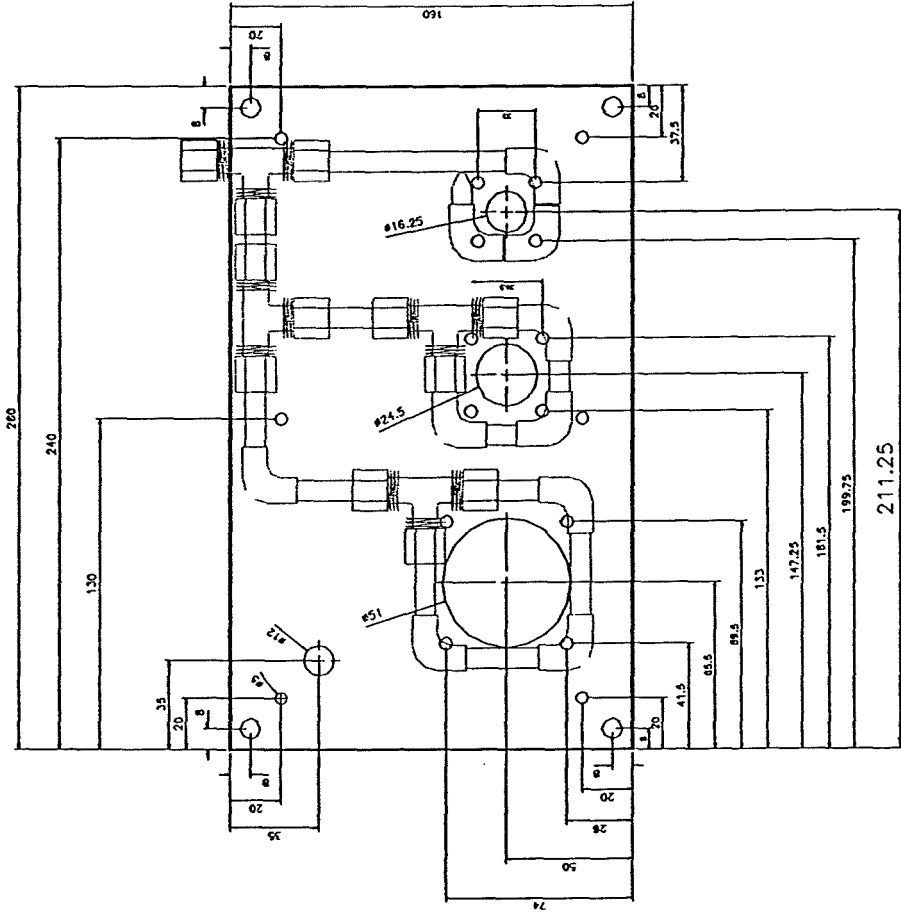




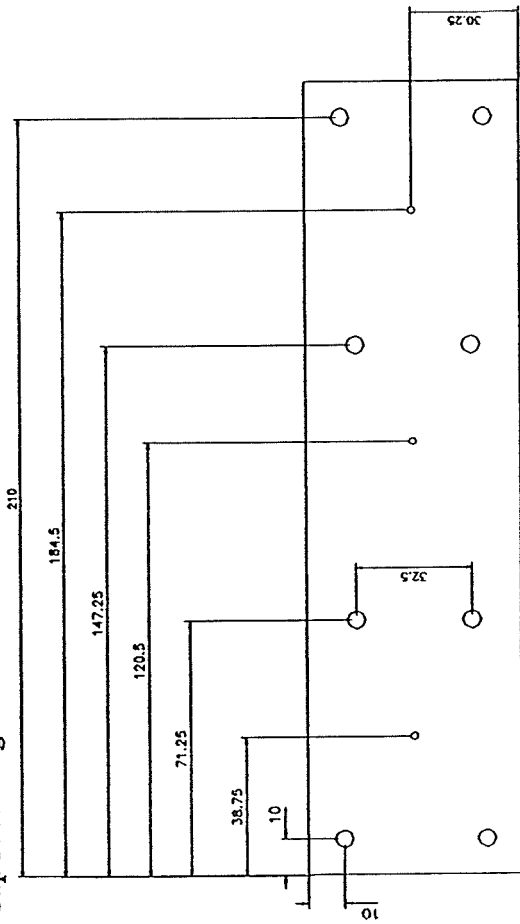
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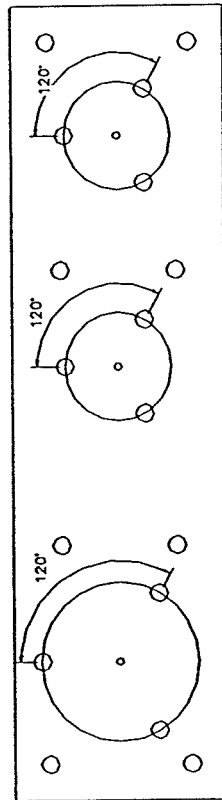
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UltraSonic Bath - Cap Mounting Plates

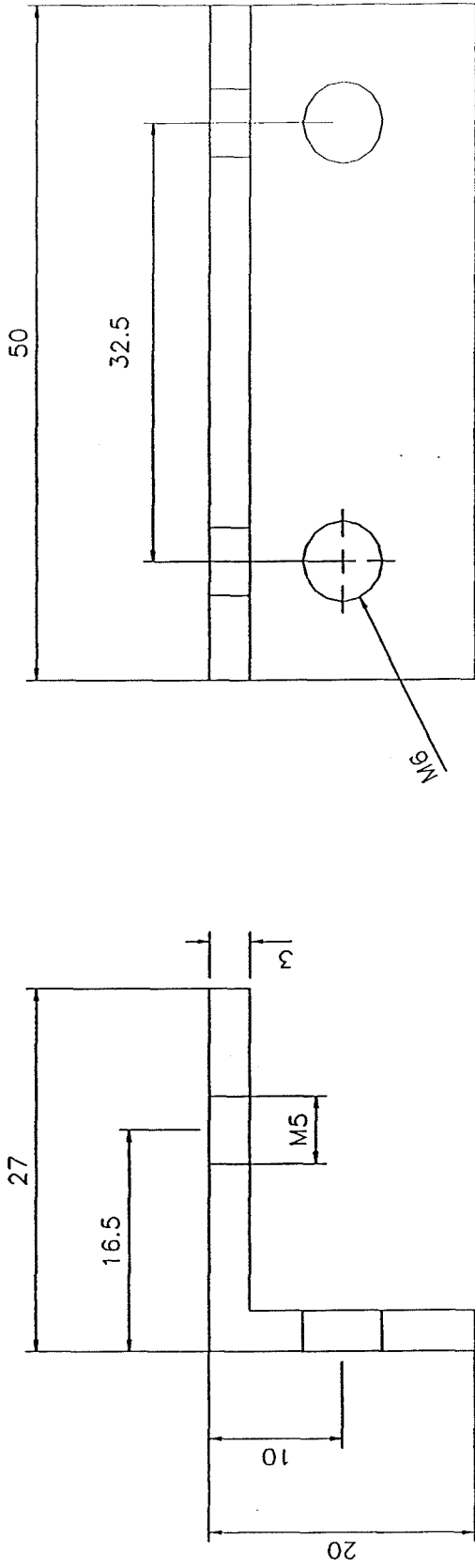


TOP PLATE



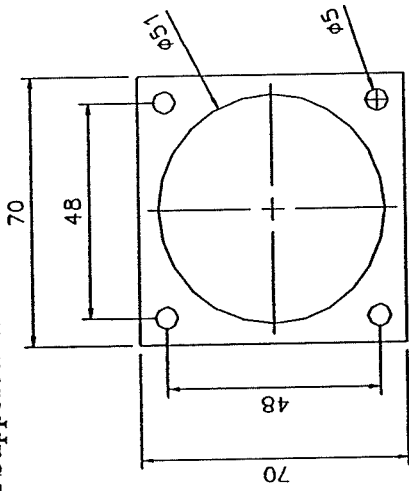
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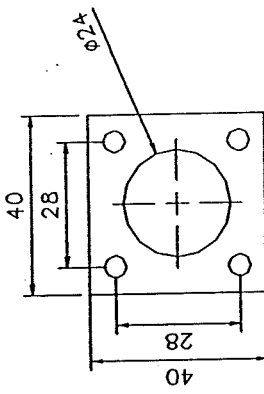




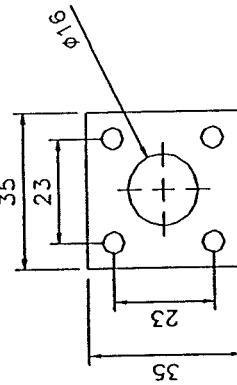
UltraSonic Bath - Vessel Support Plates



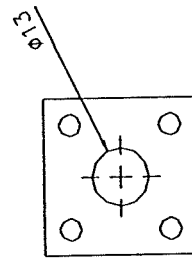
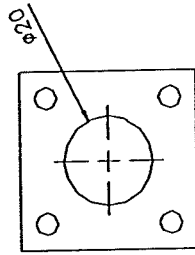
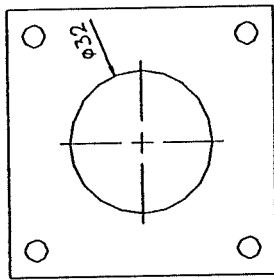
50mm tube plates



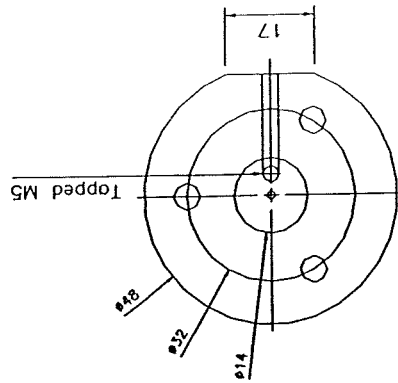
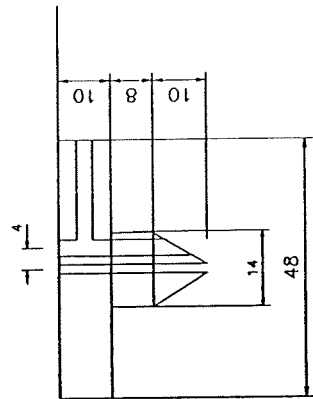
24mm tube plates



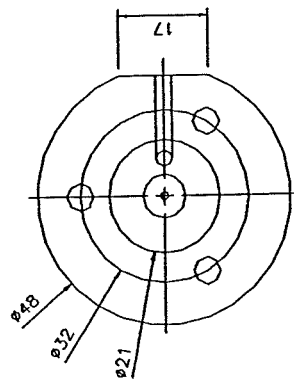
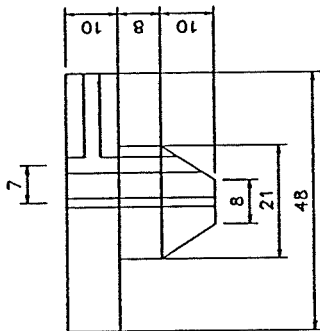
16mm tube plates



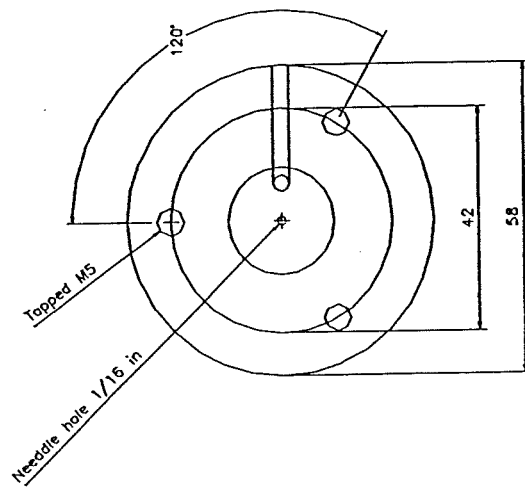
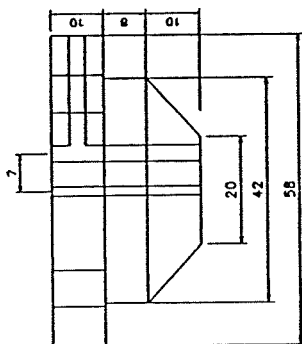
UltraSonic Bath - Caps



16mm cap

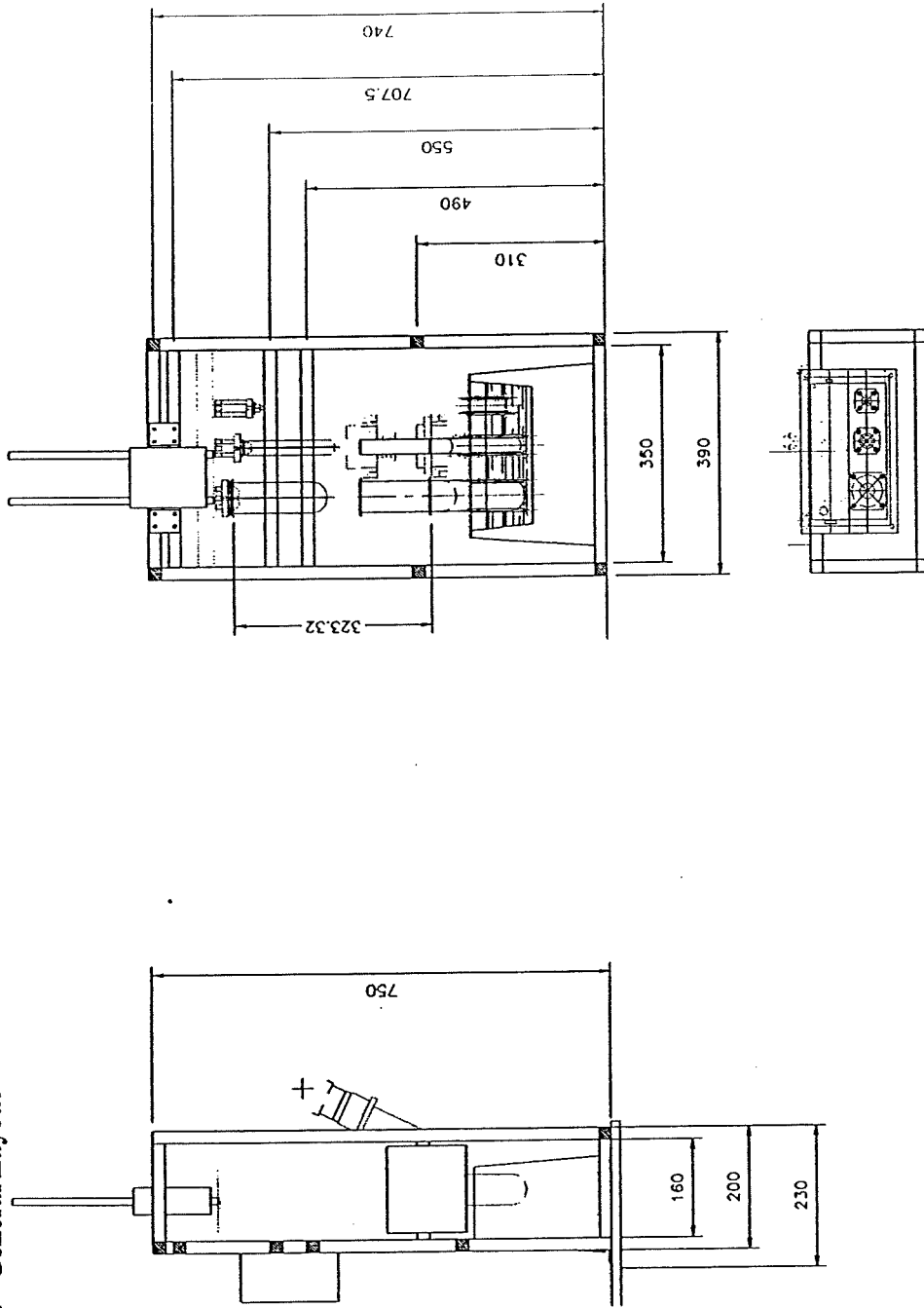


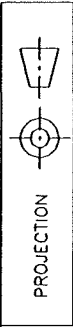
24mm cap



50mm cap

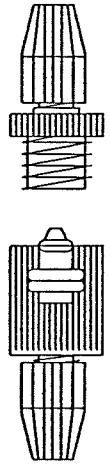
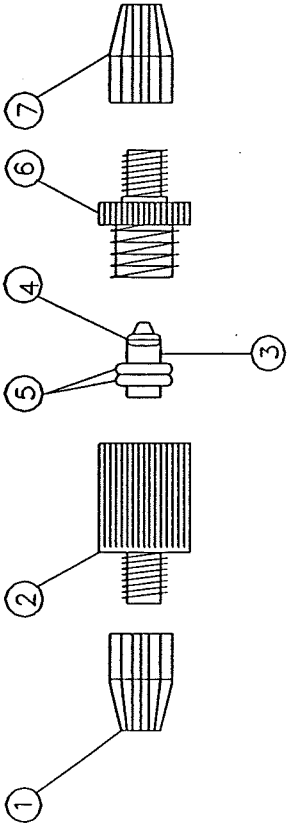
UltraSonic Bath - General Layout





DRG NO. SP02

- 1 Green 1/4'-28 - 5/16'-22 Cap
- 2 Check valve housing: Polypropylene
- 3 Plunger includes spring: 316 Stainless Steel
- 4 Brown Seal: Elastomere
- 5 Black Seal: Elastomere
- 6 Check valve housing cap: Polypropylene
- 7 Red 1/4'-28 - 5/16'-22 Cap



NOTE: Original Elastomere seals have been replaced

④ Replaced with 00-004E070 (P/N 62839) EPDM seals from Oswald Seals

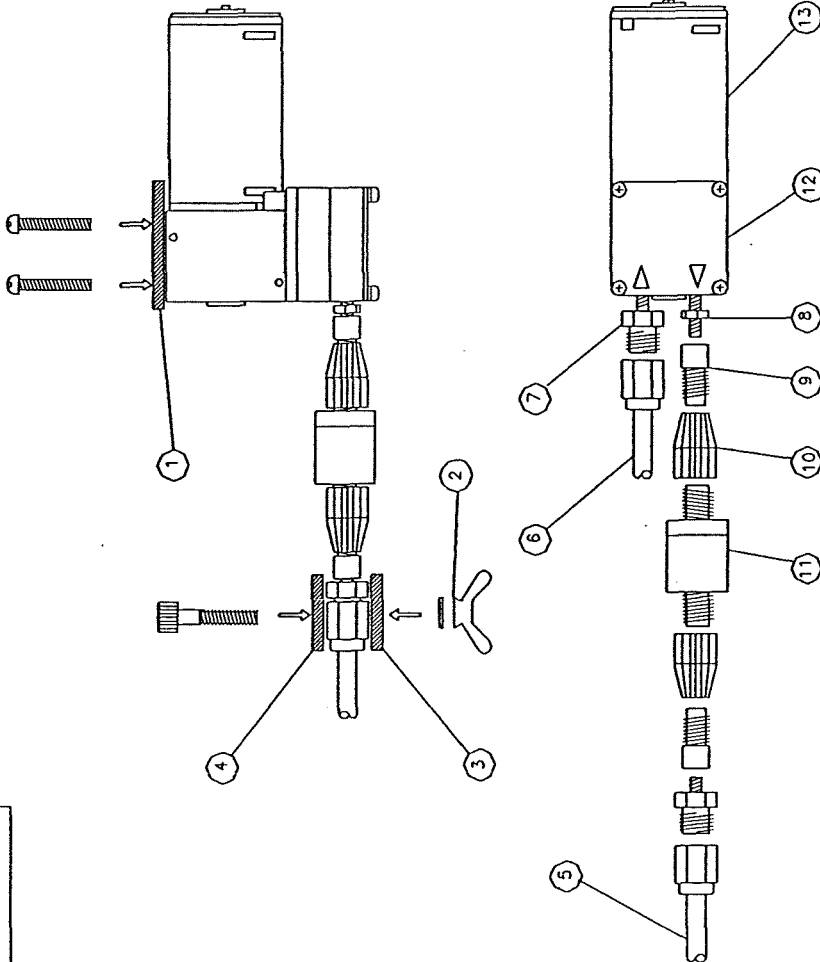
⑤ Replaced with 00-010E070 (P/N 46107) EPDM seals from Oswald Seals

TITLE: CHECK VALVE ASSEMBLY		DIMENSIONS IN MILLIMETRES (mm) TOLERANCES UNLESS OTHERWISE STATED +/- 0.1mm		Rhone-Poulenc Agriculture Ltd	
REV	DATE	MATL	FINISH	SCALE	Do not scale
		N/A			
CHANGE			DRAWN: D DUGENDRE	27 AUGUST 98	
			CHECKED:		
			FILE: DEN C:\AUTO_PRO\WORKSTAT\CHEKVALV.DWG		



PROJECTION

- 1 Pump's head attachment
- 2 Wing nut
- 3 Solvent line connection support
- 4 Wing nut support
- 5 Solvent line (to nozzles)
- 6 Solvent line (from reservoir)
- 7 Reducing Unions 1/4" - 10-32 male
- 8 10-32 UNF Male Union
- 9 1/4"-28 LC fitting
- 10 1/4"-28 - 5/16"-22 Cap
- 11 Check valve
- 12 Pump's head
- 13 Pump's rotor



DRG NO. SP01

REV	DATE	CHANGE

DIMENSIONS IN MILLIMETRES (mm)  
 TOLERANCES UNLESS OTHERWISE STATED  
 +/- 0.1mm

PHONE POULENC AGRICULTURE LTD

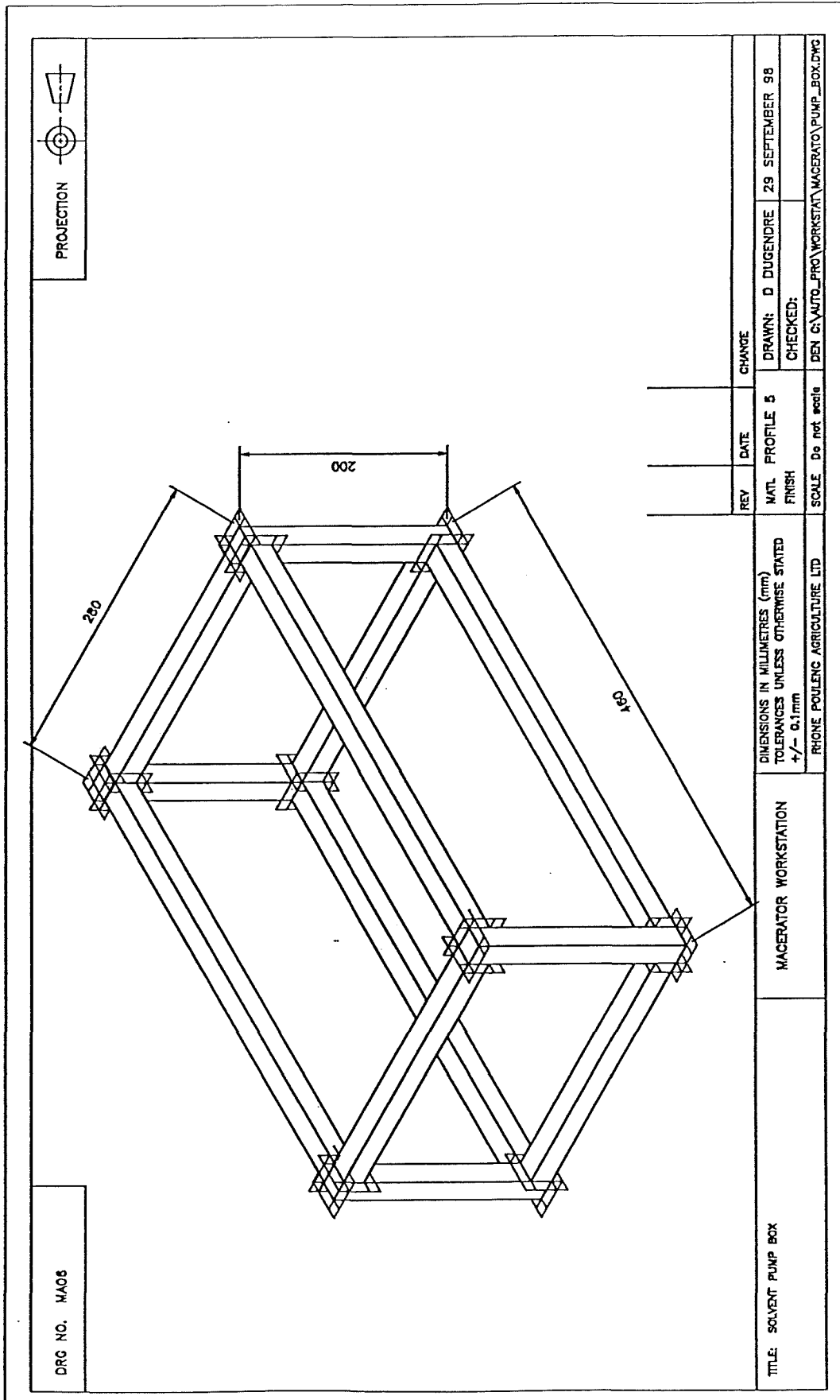
PUMP BOX

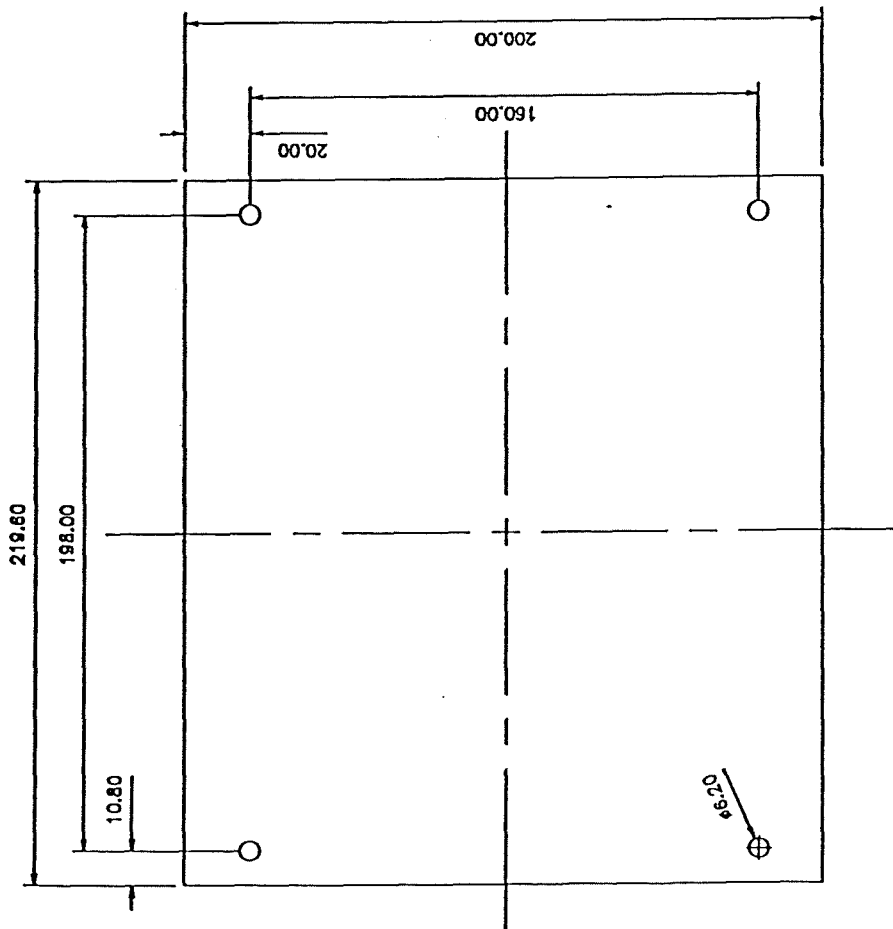
TITLE SOLVENT PUMP ASSEMBLY

SCALE Do not scale

DRAWN: D DUGENDRE  
 CHECKED:

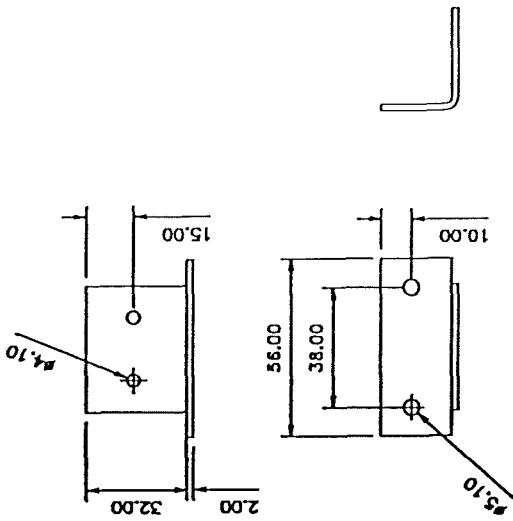
12 FEBRUARY 1998  
 FILE: DEN C:/AUTO\_PRO/WORKSTAT/KNFPUMP.DWG



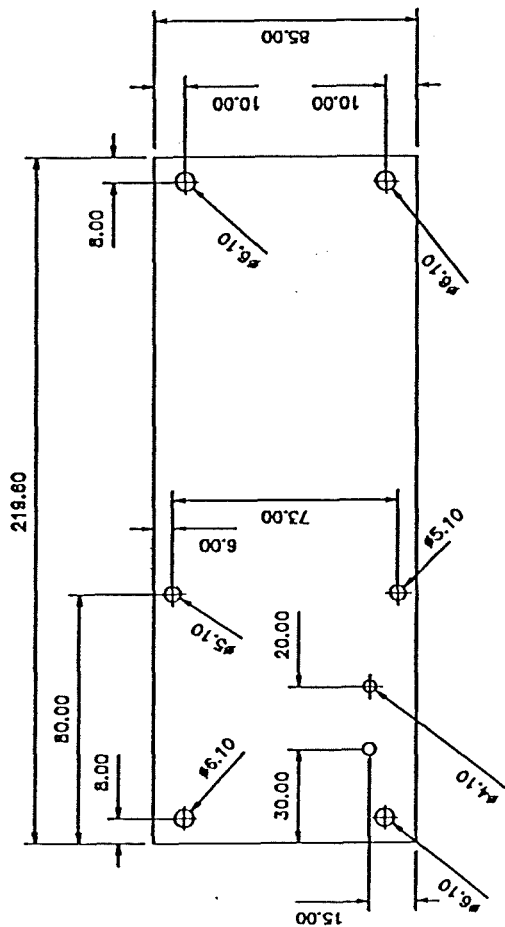


SHELF FOR STIRRER CONTROLLER  
MATERIAL: POLYPROPYLENE  
THICKNESS 6mm

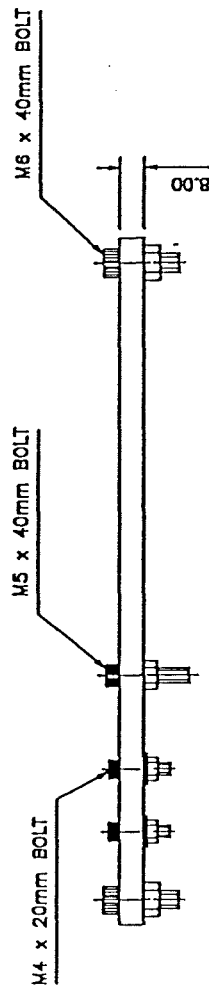
SHELF FOR STIRRER CONTROLLER	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: c:\DATA\WORKSTAT\EXTRACT\SHELFSTR.DWG	
DRAWN: N SMARTT	21 MAY 96
REVISION: 00	
RHONE POULENC AGRICULTURE LTD	



BRACKET FOR AIR REGULATOR  
MATERIAL: ALUMINIUM



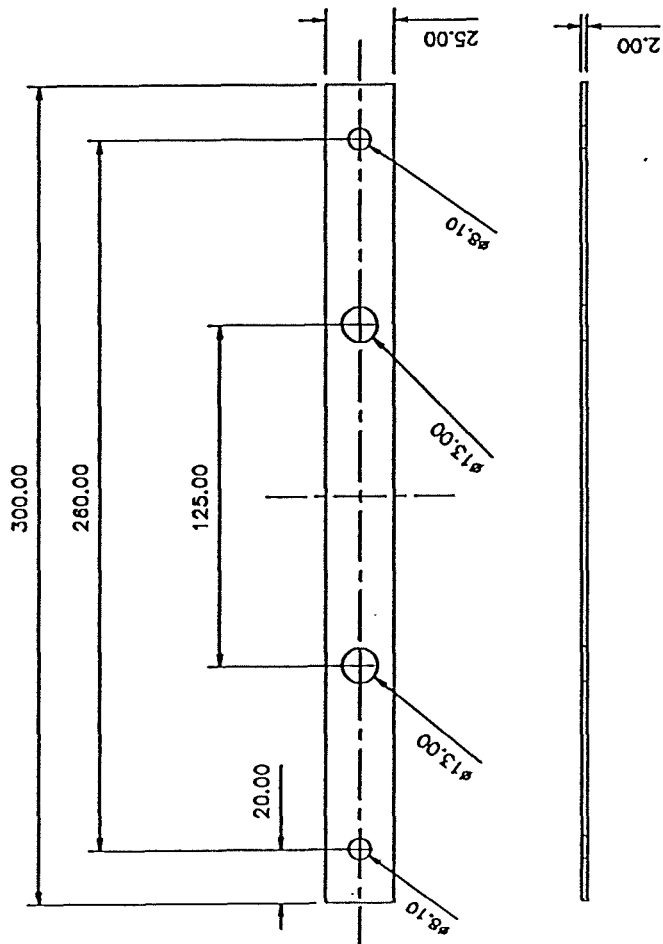
SUPPORT SHELF FOR PNEUMATIC VALVES  
MATERIAL: ALUMINIUM



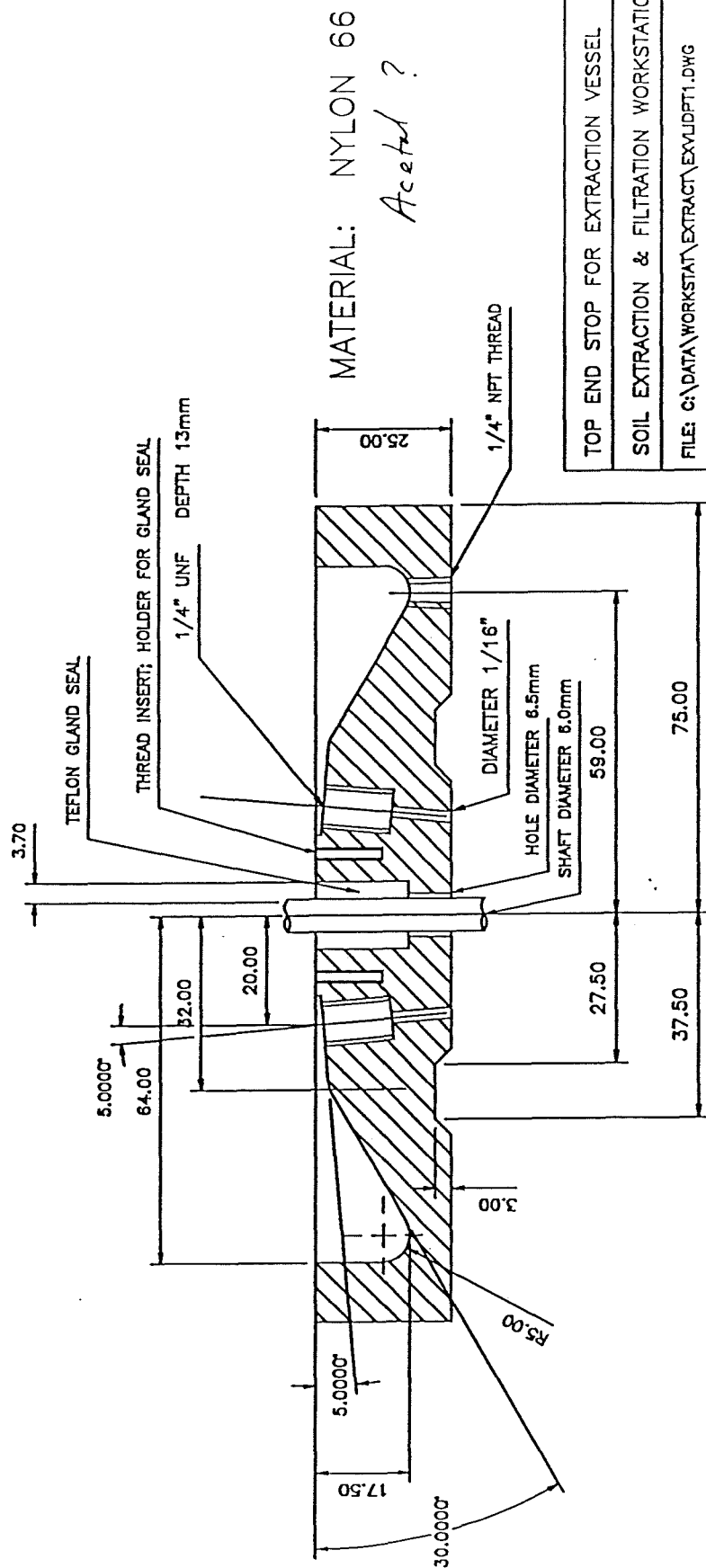
SUPPORT FOR PNEUMATIC VALVES	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\PNEUSUPP.DWG	
DRAWN: N SMARTT	22 MAY 96
REVISION: 00	
RHONE POULENC AGRICULTURE LTD	



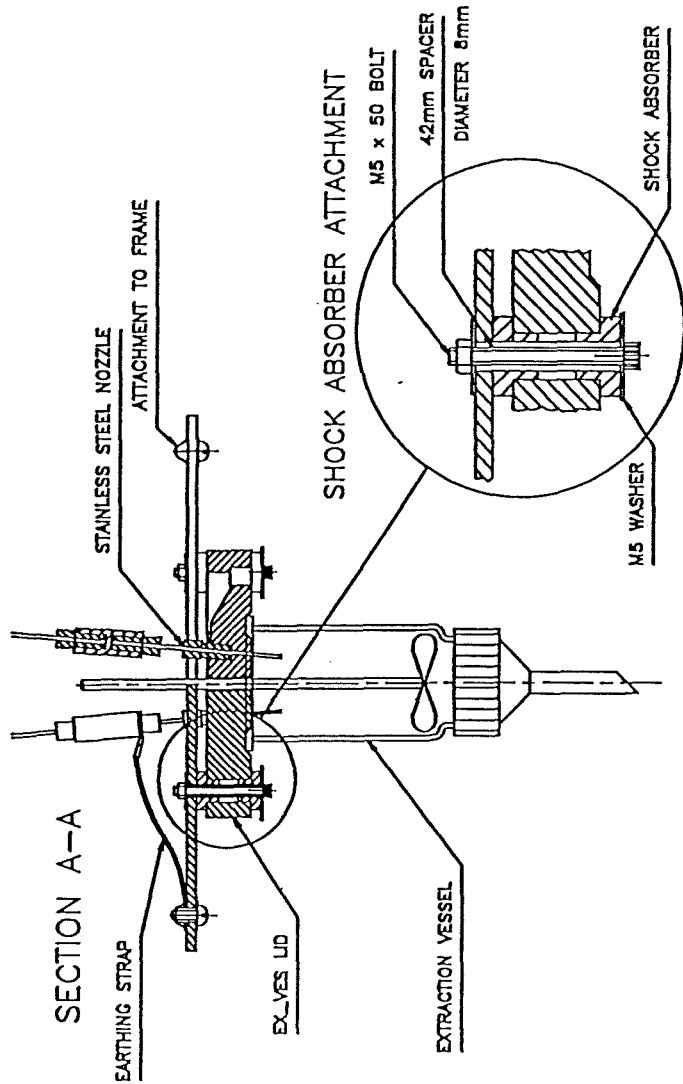
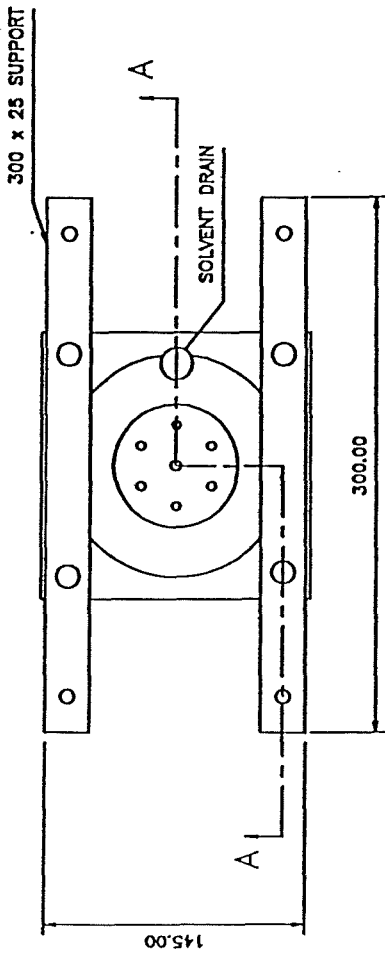
SUPPORT FOR EXTRACTION VESSEL END STOP  
MATERIAL: ALUMINIUM  
THICKNESS: APPROX 2mm  
2 OFF



SUPPORT FOR EXTRACTION VESSEL END STOP	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\LIDSUPP.DWG	
DRAWN: N SMARTT	17 MAY 96
REVISION: 00	
RHONE POULENC AGRICULTURE LTD	



TOP END STOP FOR EXTRACTION VESSEL	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\EXVUDPT1.DWG	
DRAWN: N SMARTT	20 MAY 96
REVISION: 00	
RHONE POULENC AGRICULTURE LTD	

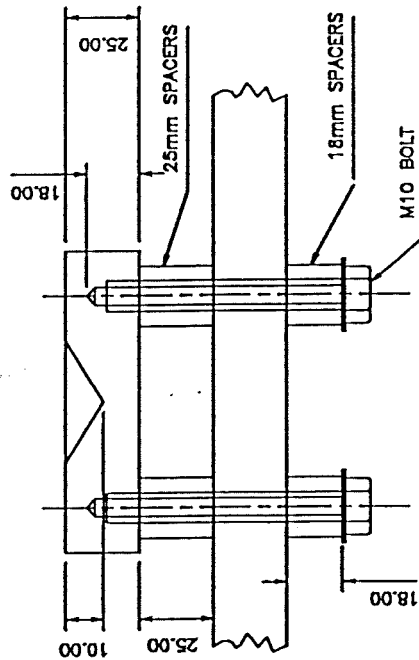
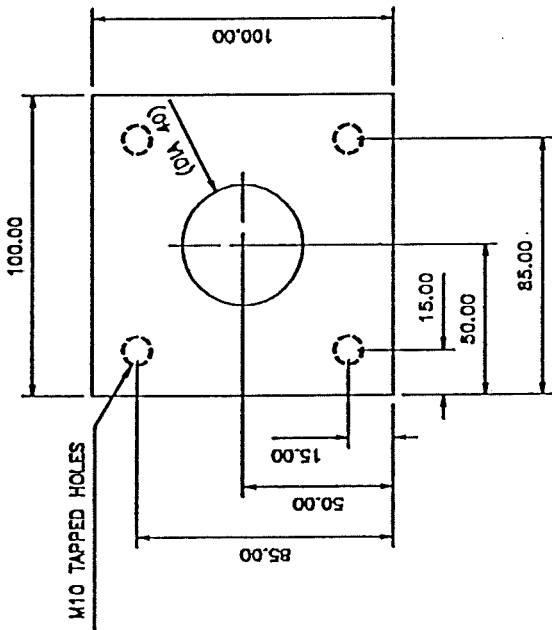


PARTS LIST

- 1 OFF EX-YES LID
- 2 OFF 300x25 SUPPORTS
- 4 OFF 42mm SPACERS
- 8 OFF SHOCK ABSORBER/VIBRATION MOUNTS
- 4 OFF M8 x 50 CAP HEAD BOLTS & NUTS
- 8 OFF DIA 30 M5 WASHERS
- 8 OFF HPLC TUBE 1/4" O.D. x 0.600 I.D. x 80mm
- 12 OFF 1/4" UNF HPLC TUBE FITTINGS
- 8 OFF 1/4" UNF MALE/MALE ADAPTOR
- 8 OFF 1/4" UNF BLANKING PLUGS
- 4 OFF EARTHING STRAP
- 4 OFF M8 x 10mm BOLTS
- 4 OFF T-SLOT NUTS M8

MATERIAL	RS PART NO
NYLON 66	688-284
ALUMINIUM	
STAINLESS STEEL	
STAINLESS STEEL	
STAINLESS STEEL	
POLYPROPYLENE	582-588

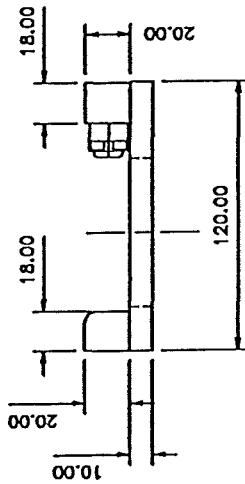
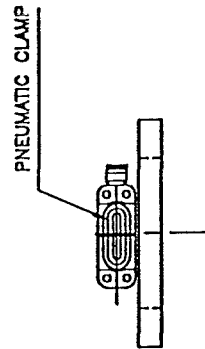
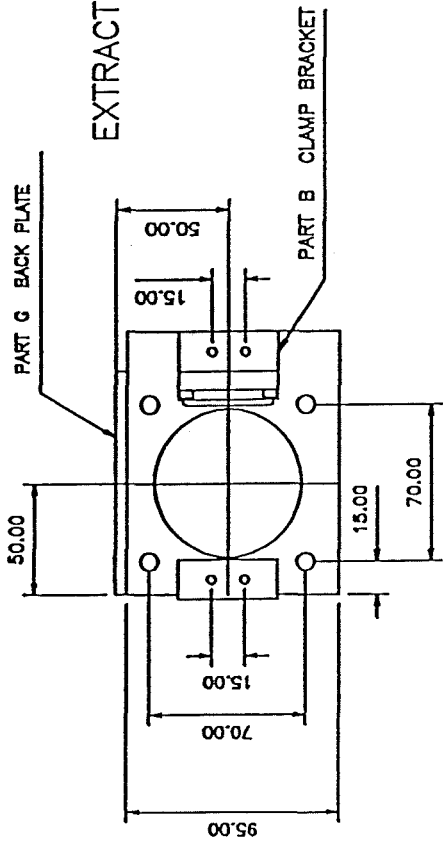
TOP END STOP FOR EXTRACTION VESSEL	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\EXYSLID.DWG	
DRAWN: N SMARTT	17 MAY 96
REVISION: 00	
RHONE POULENC AGRICULTURE LTD	



BENCH THICKNESS 1" [25.4mm]

BASE SUPPORT FOR COLLECTION VESSEL
FILE: C:\DATA\WORKSTAT\BASESUPP.DWG
MATERIAL: NYLON 66
DRAWN: N SMARTT      24 APRIL 96
REVISION: 01    9 MAY 96
RHONE POULENC AGRICULTURE LTD

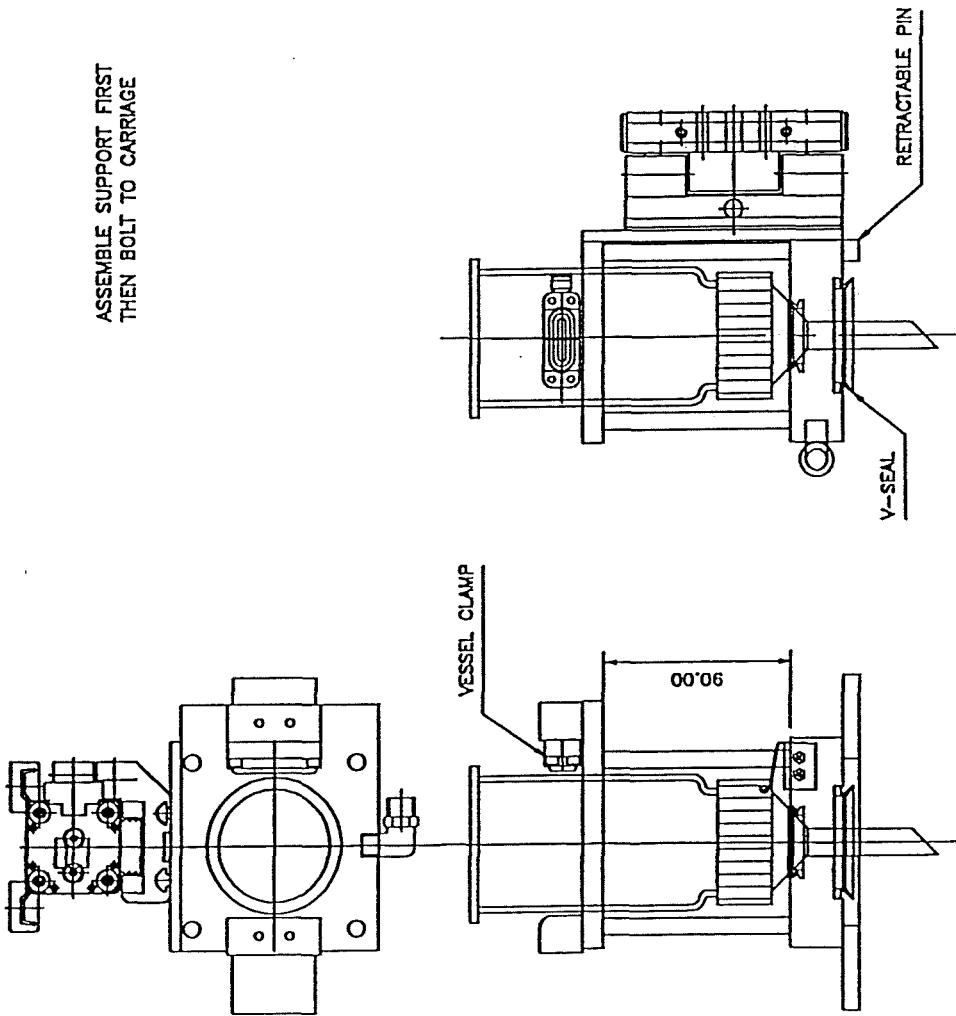
EXTRACTION VESSEL SUPPORT -- PART A  
UPPER SUPPORT



EXTRACTION VESSEL SUPPORT	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\EXVESFTA.DWG	
DRAWN: N SMARTT	05 JUNE 96
REVISION: 00	
RHONE POULENC AGRICULTURE LTD	

EXTRACTION VESSEL SUPPORT - ASSEMBLY DRAWING

ASSEMBLE SUPPORT FIRST  
THEN BOLT TO CARRIAGE

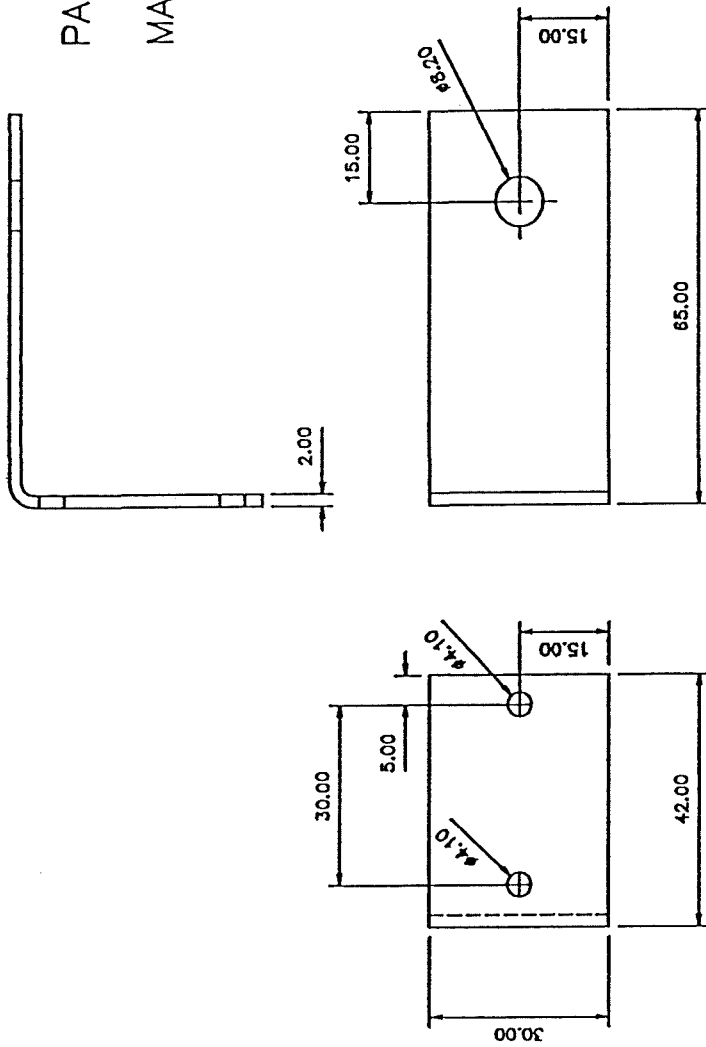


PARTS LIST

- PART A UPPER SUPPORT
- PART B CLAMP BRACKET
- PART C REACTION TO CLAMP
- PART D PNEUMATIC CLAMP
- PART E SPACER TUBES
- PART F LOWER SUPPORT
- PART G BACK PLATE
- 50mm V-RING SEAL
- 30mm V-RING SEAL
- LIMIT SWITCH
- LIMIT SWITCH BRACKET
- PNEUMATIC CLAMP ATTACHMENT BOLTS
- CLAMP BRACKET ATTACHMENT BOLTS
- MAIN THROUGH BOLTS
- VACUUM FITTING
- RETRACTABLE PIN
- SPRING
- RETAINING COLLAR
- SUPPORT- BACK PLATE ATTACHMENT BOLTS
- BACK PLATE- CARRIAGE ATTACHMENT BOLTS
- T-SLOT NUT

EXTRACTION VESSEL SUPPORT	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\EXVSSUP.DWG	
DRAWN: N SMARTT	23 MAY 96
REVISION: 00	
RHONE POULENC AGRICULTURE LTD	

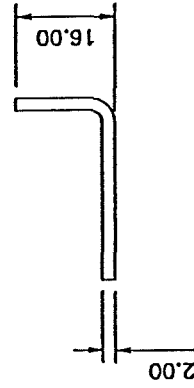
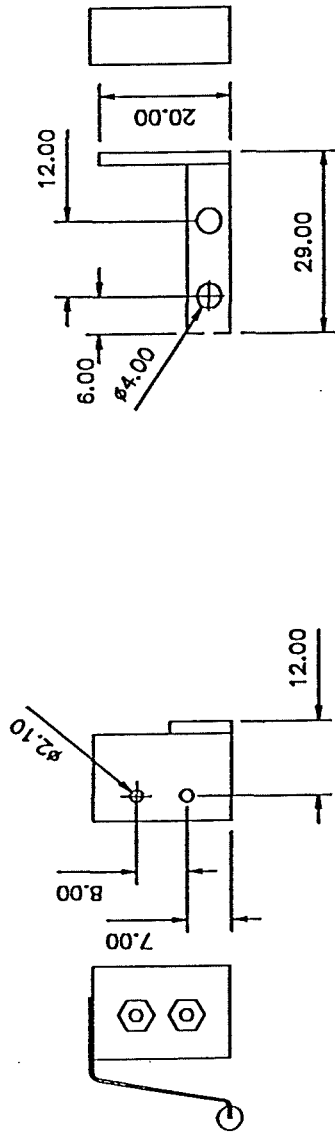
PART F: CHAIN LINK ATTACHMENT  
 MATERIAL: ALUMINIUM



SUPPORT BRACKET FOR COLLECTION VESSEL	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\CHAINBK.DWG	
DRAWN: N SMARTT	22 MAY 96
REVISION: 00	
RHONE POULENC AGRICULTURE LTD	

# PART E: LIMIT SWITCH ATTACHMENT

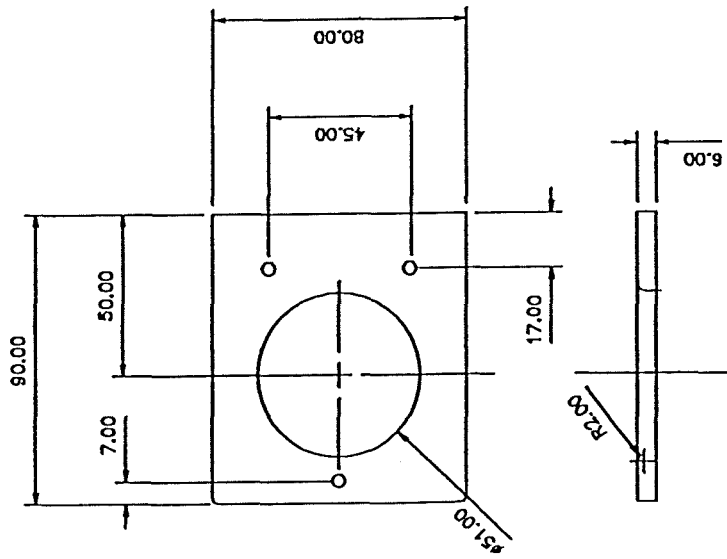
MATERIAL: ALUMINIUM



SUPPORT BRACKET FOR COLLECTION VESSEL	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\COLBRAKE.DWG	
DRAWN: N SMARTT	06 APRIL 96
REVISION: 02	23 APRIL 96
RHONE POULENC AGRICULTURE LTD	

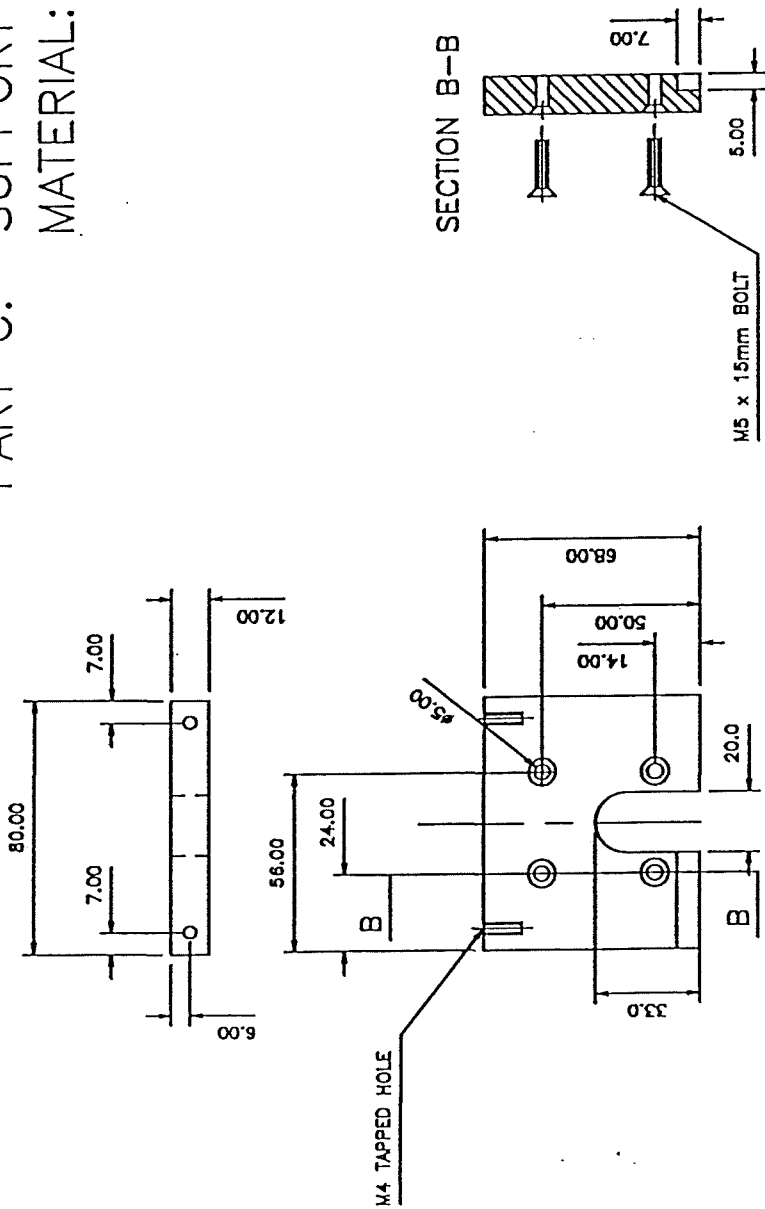


PART D: SUPPORT COVER  
MATERIAL: POLYPROPYLENE



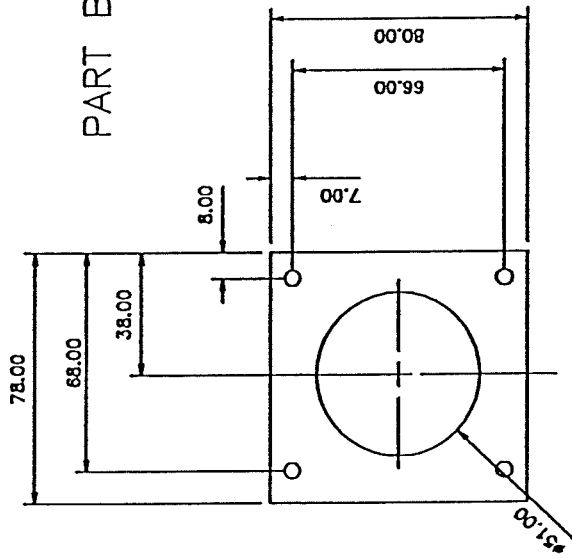
SUPPORT BRACKET FOR COLLECTION VESSEL	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\COLBRAXD.DWG	
DRAWN: N SMARTT	06 APRIL 96
REVISION: 02	23 APRIL 96
RHONE POULENC AGRICULTURE LTD	

PART C: SUPPORT BRACKET MOUNTING  
 MATERIAL: ALUMINIUM

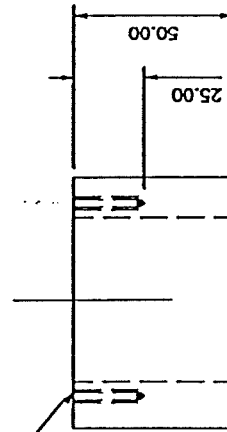


SUPPORT BRACKET FOR COLLECTION VESSEL	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\COLBRACK.DWG	
DRAWN: N SMARTT	06 APRIL 96
REVISION: 02	23 APRIL 96
RHONE POULENC AGRICULTURE LTD	

PART B: LOWER VESSEL SUPPORT  
 MATERIAL: NYLON 66

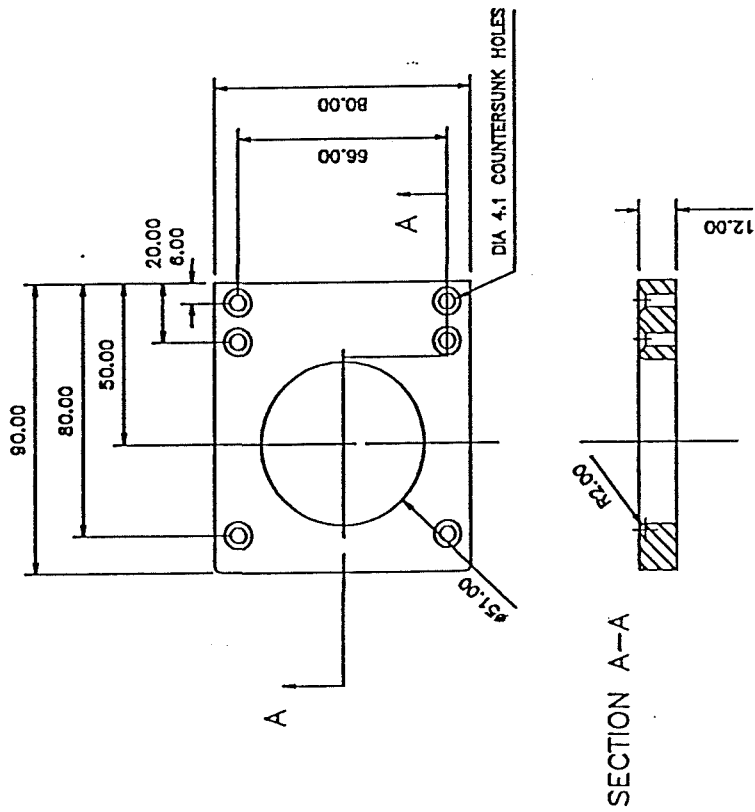


M4 TAPPED HOLE



SUPPORT BRACKET FOR COLLECTION VESSEL	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\COLBRAKE.DWG	
DRAWN: N SMARTT	06 APRIL 96
REVISION: 02	23 APRIL 96
RHONE POULENC AGRICULTURE LTD	

PART A: UPPER VESSEL SUPPORT  
MATERIAL: ALUMINIUM



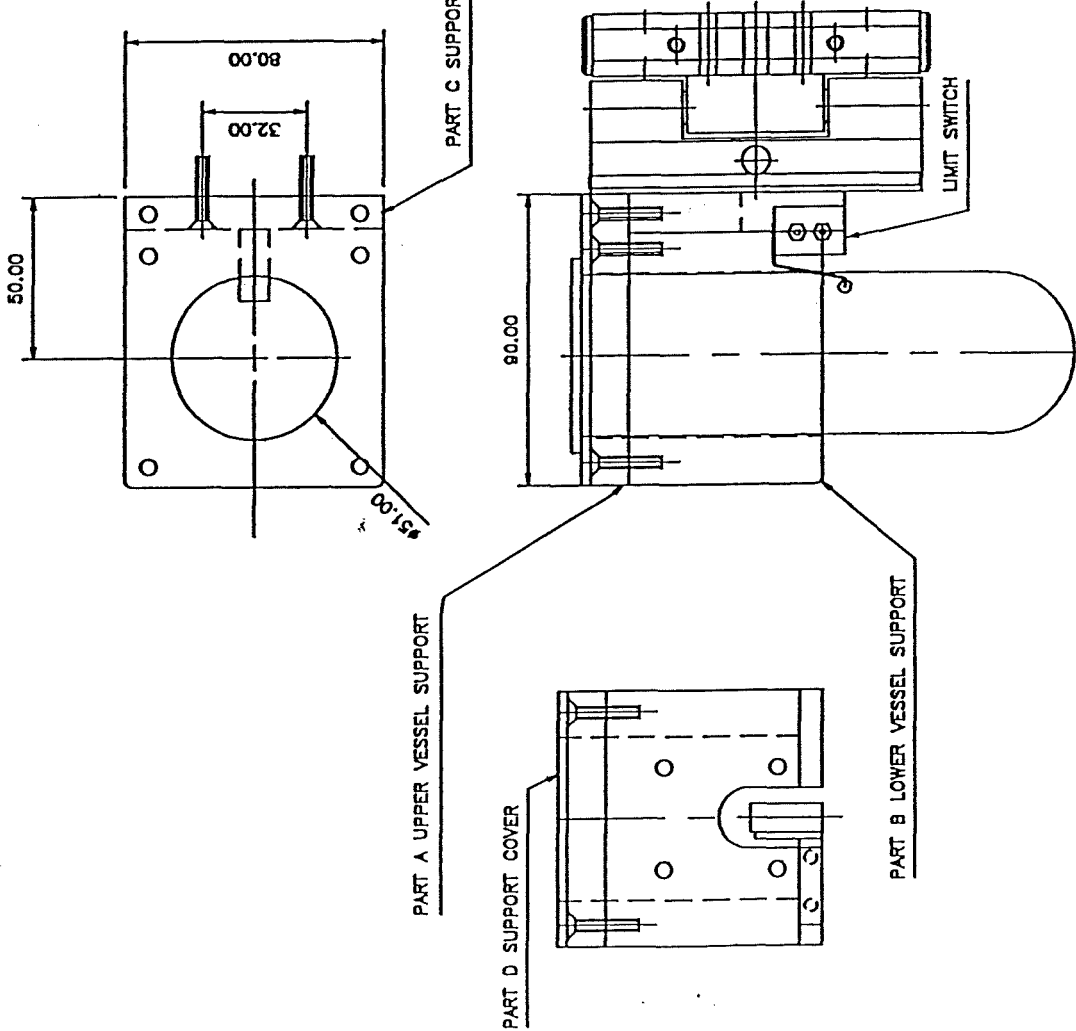
SUPPORT BRACKET FOR COLLECTION VESSEL	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: G:\DATA\WORKSTAT\EXTRACT\COLBRAKA.DWG	
DRAWN: N SMARTT	06 APRIL 96
REVISION: 02	23 APRIL 96
RHONE POULENC AGRICULTURE LTD	

PARTS LIST

- ALUMINIUM
- NYLON 66
- ALUMINIUM
- POLYPROPYLENE
- ALUMINIUM

PARTS LIST

- PART A UPPER VESSEL SUPPORT
- PART B LOWER VESSEL SUPPORT
- PART C SUPPORT BRACKET MOUNTING
- PART D SUPPORT COVER
- LIMIT SWITCH RS PART NO 334-870
- LIMIT SWITCH BRACKET
- CABLE TRUNKING BRACKET
- ROUND HEAD SCREWS
- M4 x 20 COUNTERSUNK SCREWS

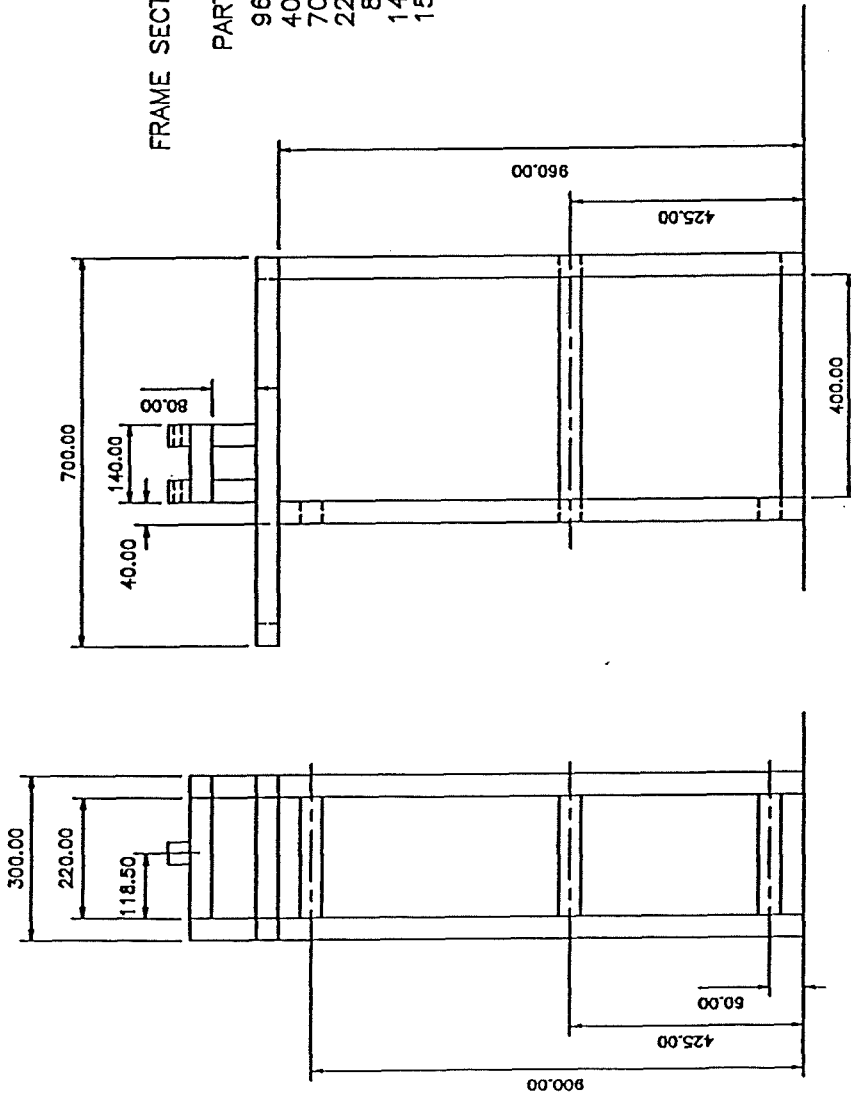


SUPPORT FOR COLLECTION VESSEL	
SOIL EXTRACTION & FILTRATION WORKSTATION	
FILE: C:\DATA\WORKSTAT\EXTRACT\COL_ASB.DWG	
DRAWN: N SMARTT	06 APRIL 96
REVISION: 02	23 APRIL 96
RHONE POULENC AGRICULTURE LTD	

FRAME SECTIONS FROM ITEM ECONOMY SECTION

PARTS LIST NO REQUIRED

960mm	4
400mm	4
700mm	2
220mm	9
80mm	4
140mm	2
150mm	2



FRAME FOR STIRRER WORKSTATION

SOIL EXTRACTION & FILTRATION WORKSTATION

FILE: C:\DATA\WORKSTAT\EXTRACT\STRFRAME.DWG

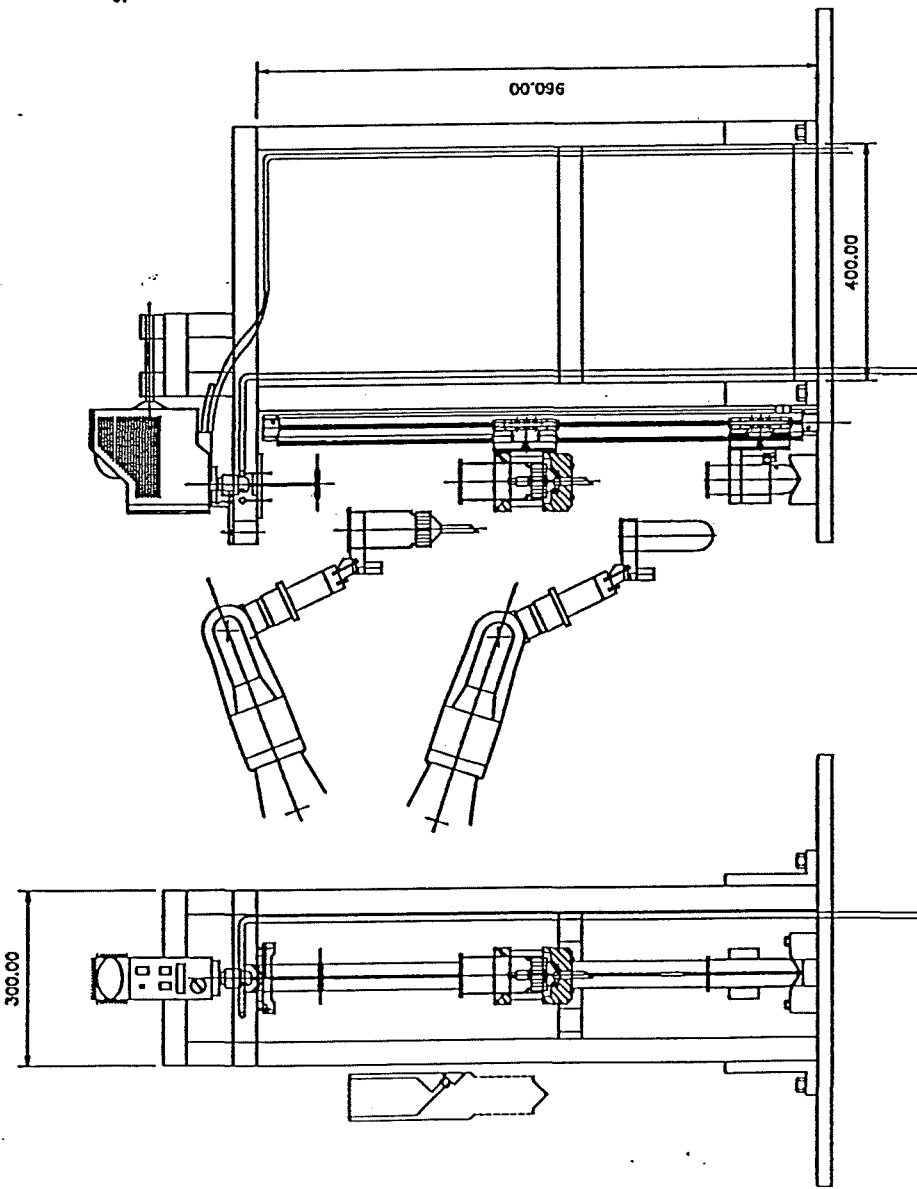
DRAWN: N SMARTT APRIL 96

REVISION: 00

RHONE POULENC AGRICULTURE LTD

SUB ASSEMBLIES

- FRAME
- PNEUMATIC RODLESS CYLINDER
- COLLECTION VESSEL HOLDER
- EXTRACTION VESSEL HOLDER
- TOP END STOP
- BOTTOM END STOP
- HEIDOLPH STIRRER BRACKET
- STIRRER CONTROLLER SUPPORTS
- PNEUMATIC VALVE SUPPORTS
- SOLVENT DELIVERY SYSTEM



SOIL EXTRACTION & FILTRATION WORKSTATION

GENERAL ARRANGEMENT

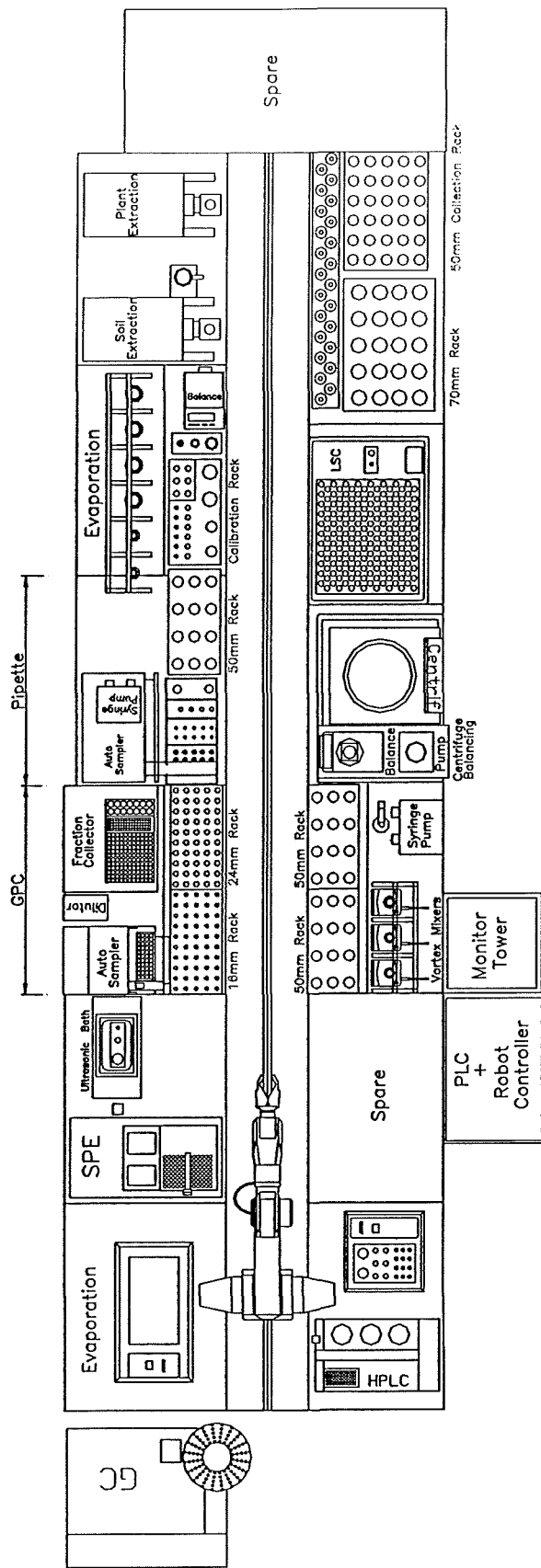
FILE: C:\DATA\WORKSTAT\EXTRACT\STIR1005.DWG

DRAWN: N SMARTT 24 APRIL 96

REVISION: 02 10 MAY 96

RHONE POULENC AGRICULTURE LTD

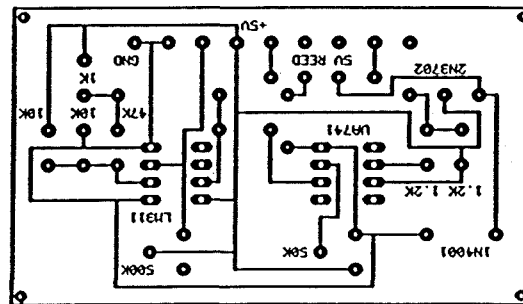
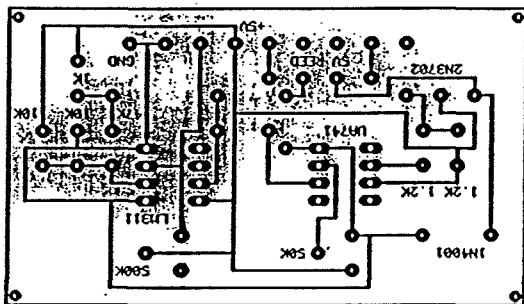
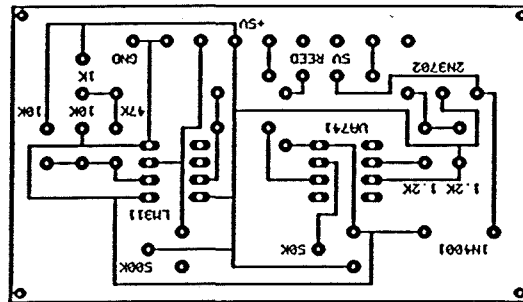
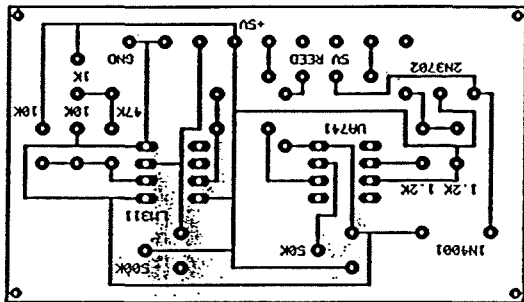
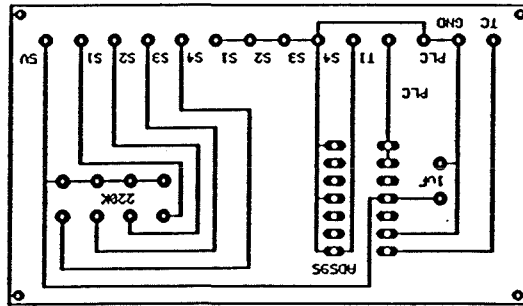
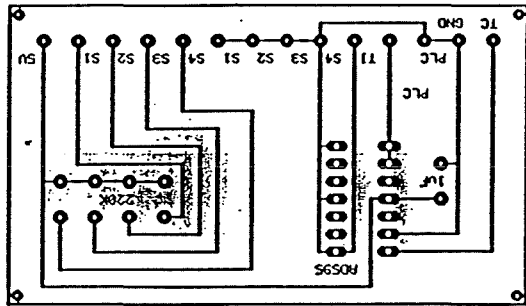
# System Layout

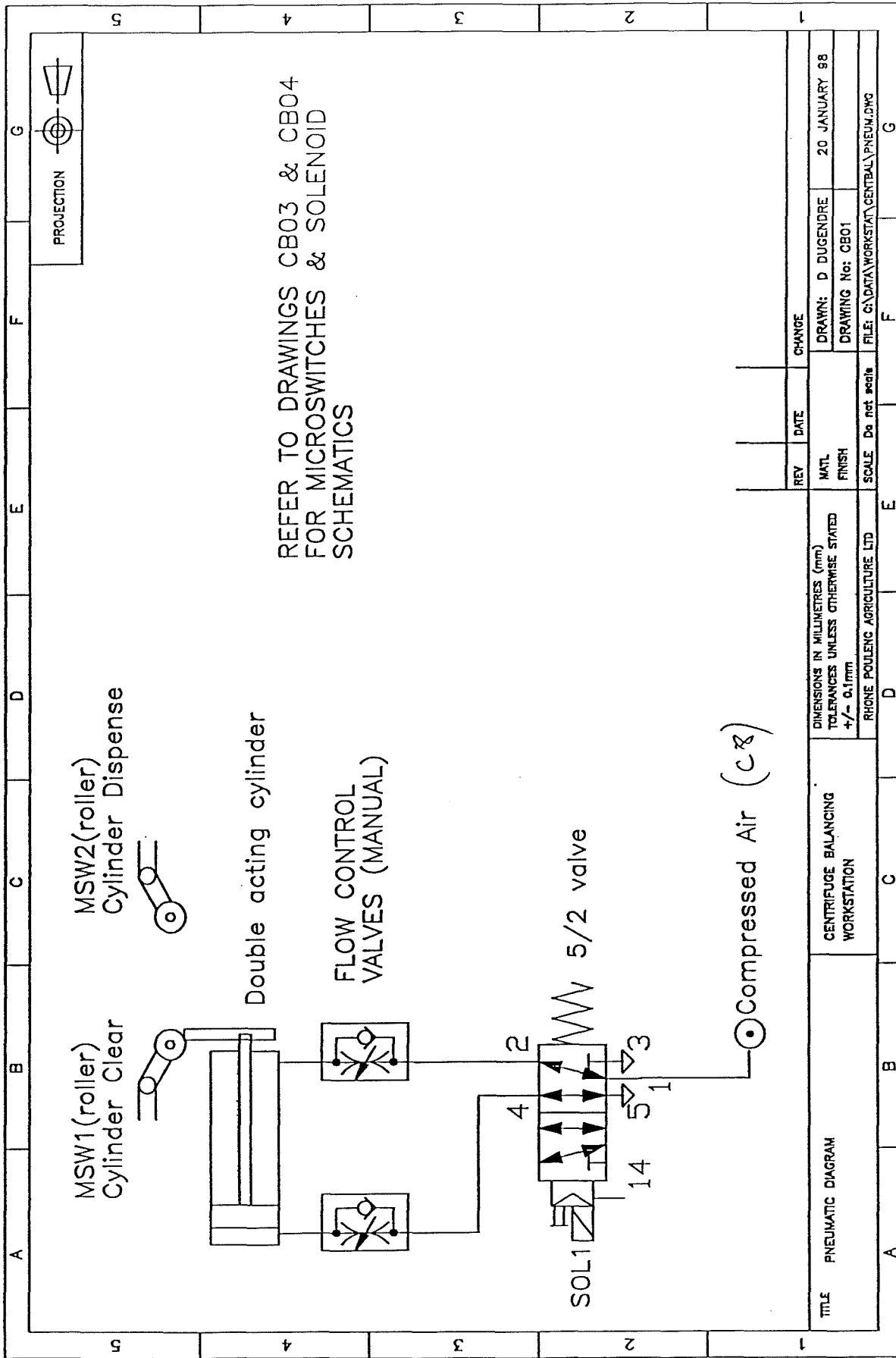




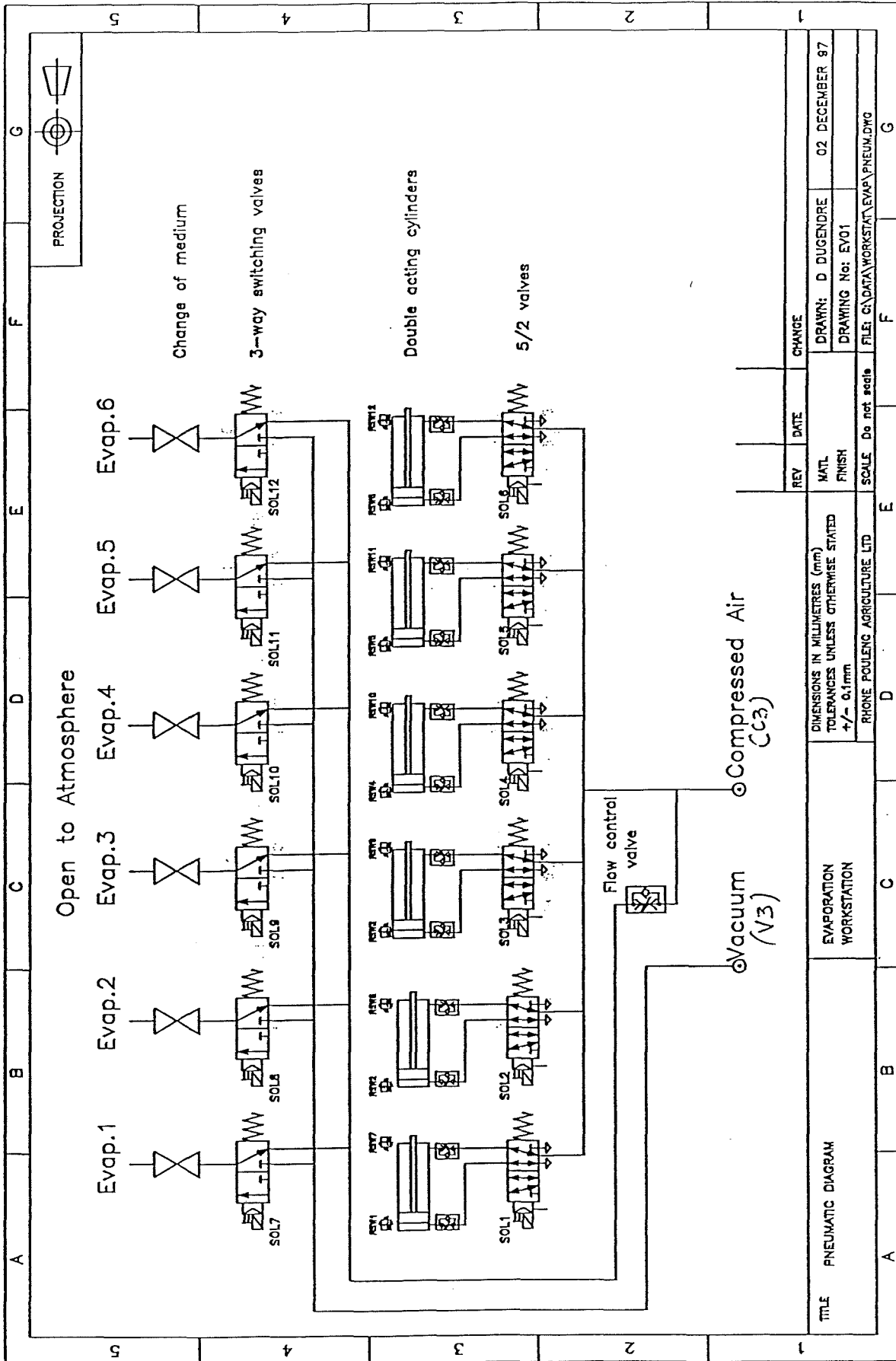
### 10.3. Appendix C: Electrical & Pneumatic Drawings

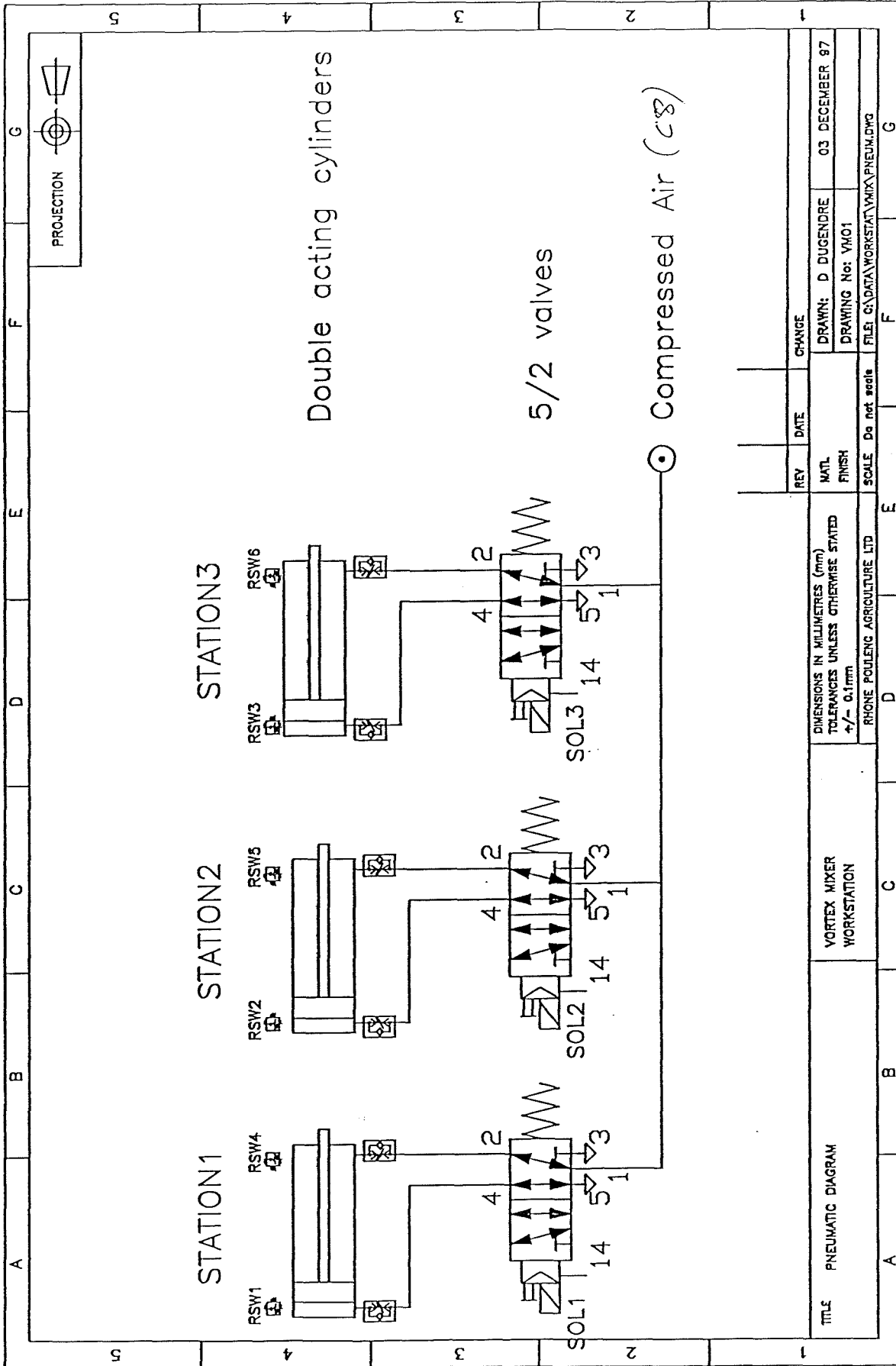
PCB Layout of evaporation workstation - Liquid Level Detection and Temperature Sensing



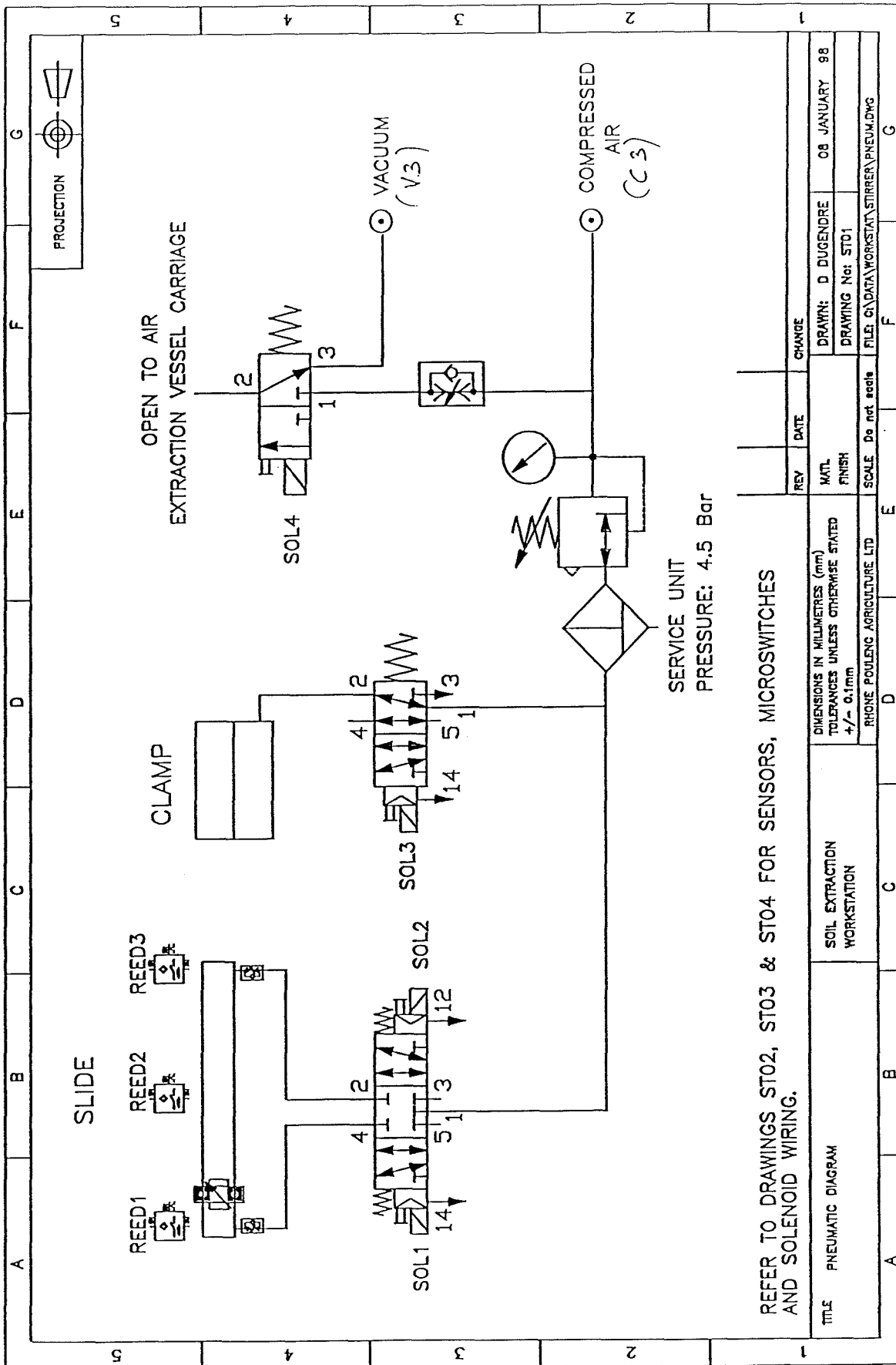






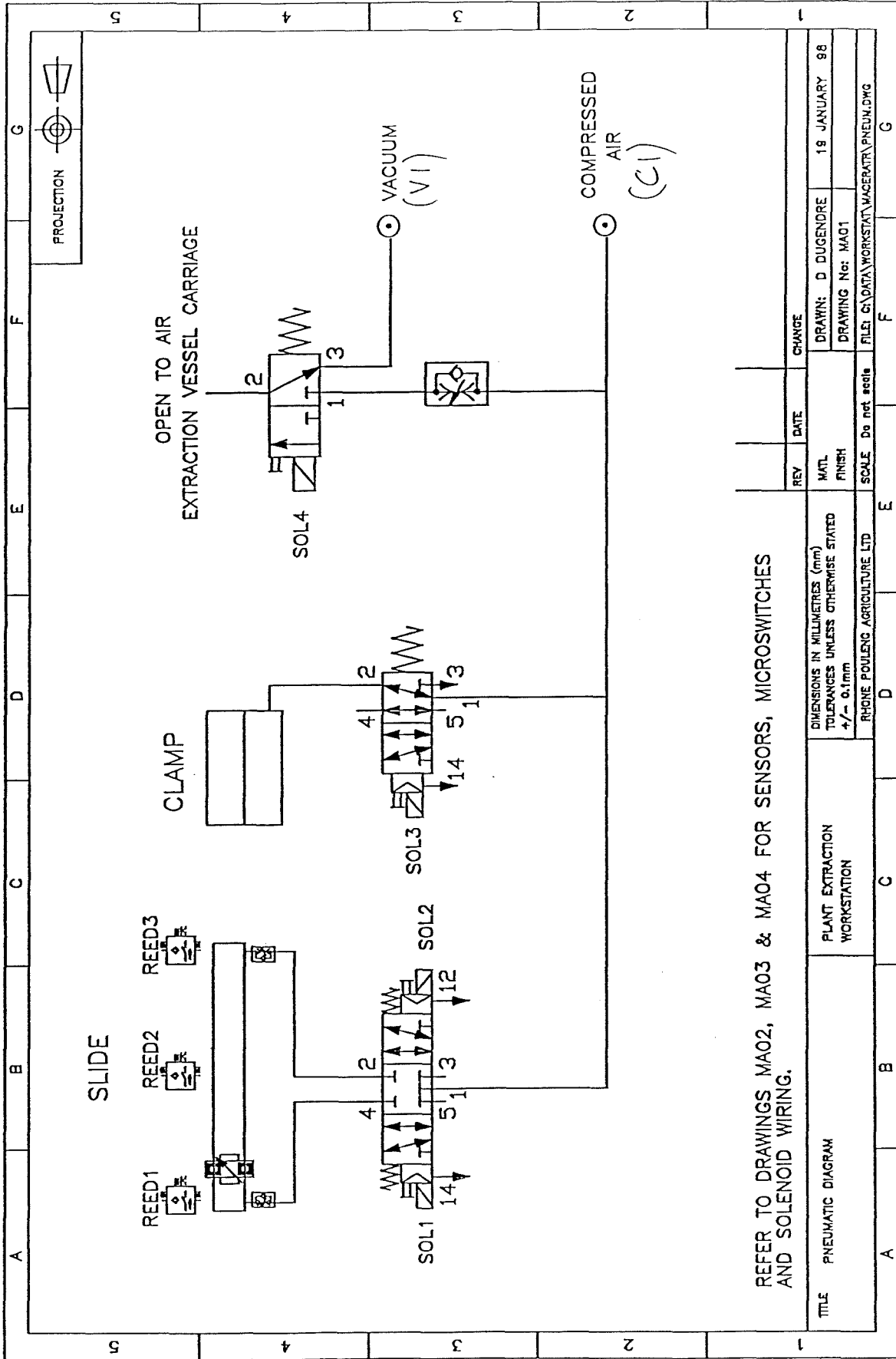


REF	DATE	CHANGE
DIMENSIONS IN MILLIMETRES (mm)		
TOLERANCES UNLESS OTHERWISE STATED		
+/- 0.1mm		
RHONE POULENC AGRICULTURE LTD		
VORTEX MIXER WORKSTATION		
PNEUMATIC DIAGRAM		
DRAWN: D DUGENDRE		
DRAWING No: VMO1		
03 DECEMBER 97		
FILE: G:\DATA\WORKSTAT\VMIX\FRUM.DWG		



REFER TO DRAWINGS ST02, ST03 & ST04 FOR SENSORS, MICROSWITCHES AND SOLENOID WIRING.

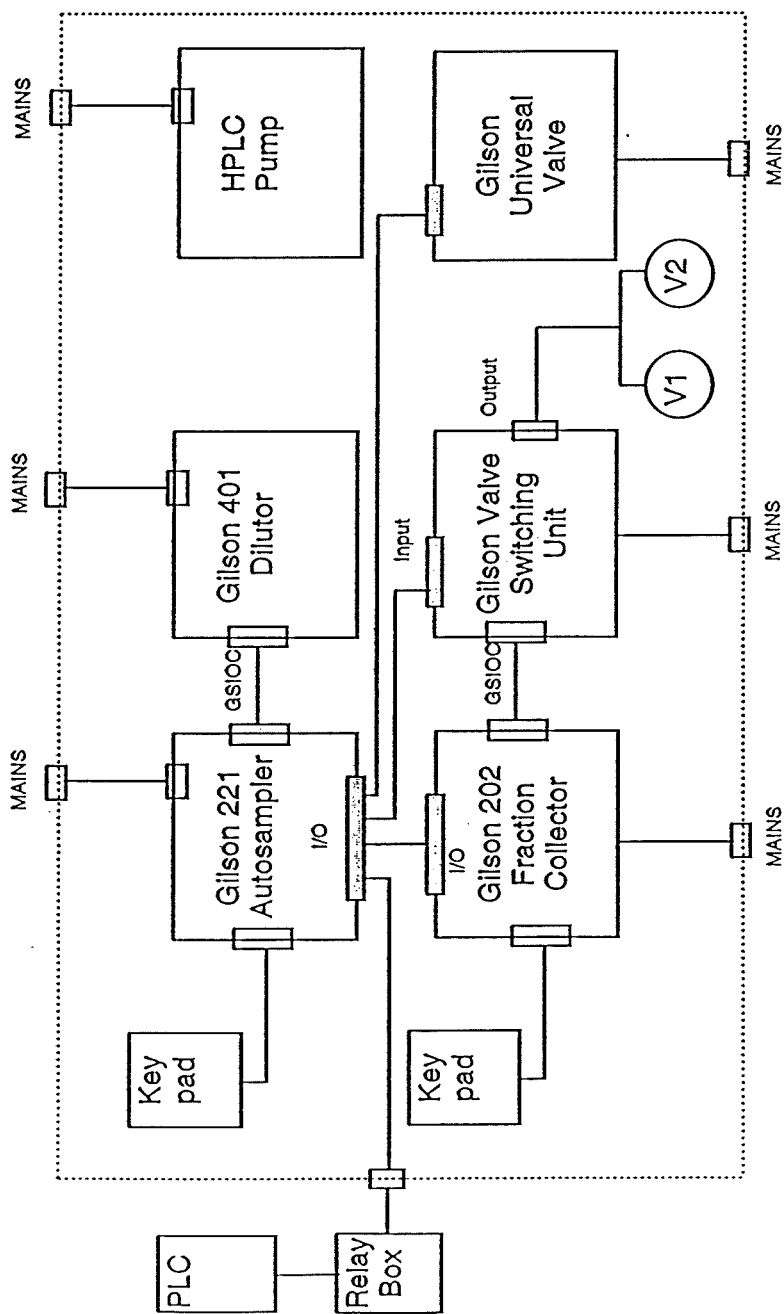
FILE	PNEUMATIC DIAGRAM	SOIL EXTRACTION WORKSTATION	DIMENSIONS IN MILLIMETRES (mm) TOLERANCES UNLESS OTHERWISE STATED +/- 0.1mm	REV	DATE	CHANGE
				MATL	08 JANUARY 98	
				FINISH		
				SCALE	Do not scale	
						FILE: G:\DATA\WORKSTAT\STIRREA\PNEUM.DWG



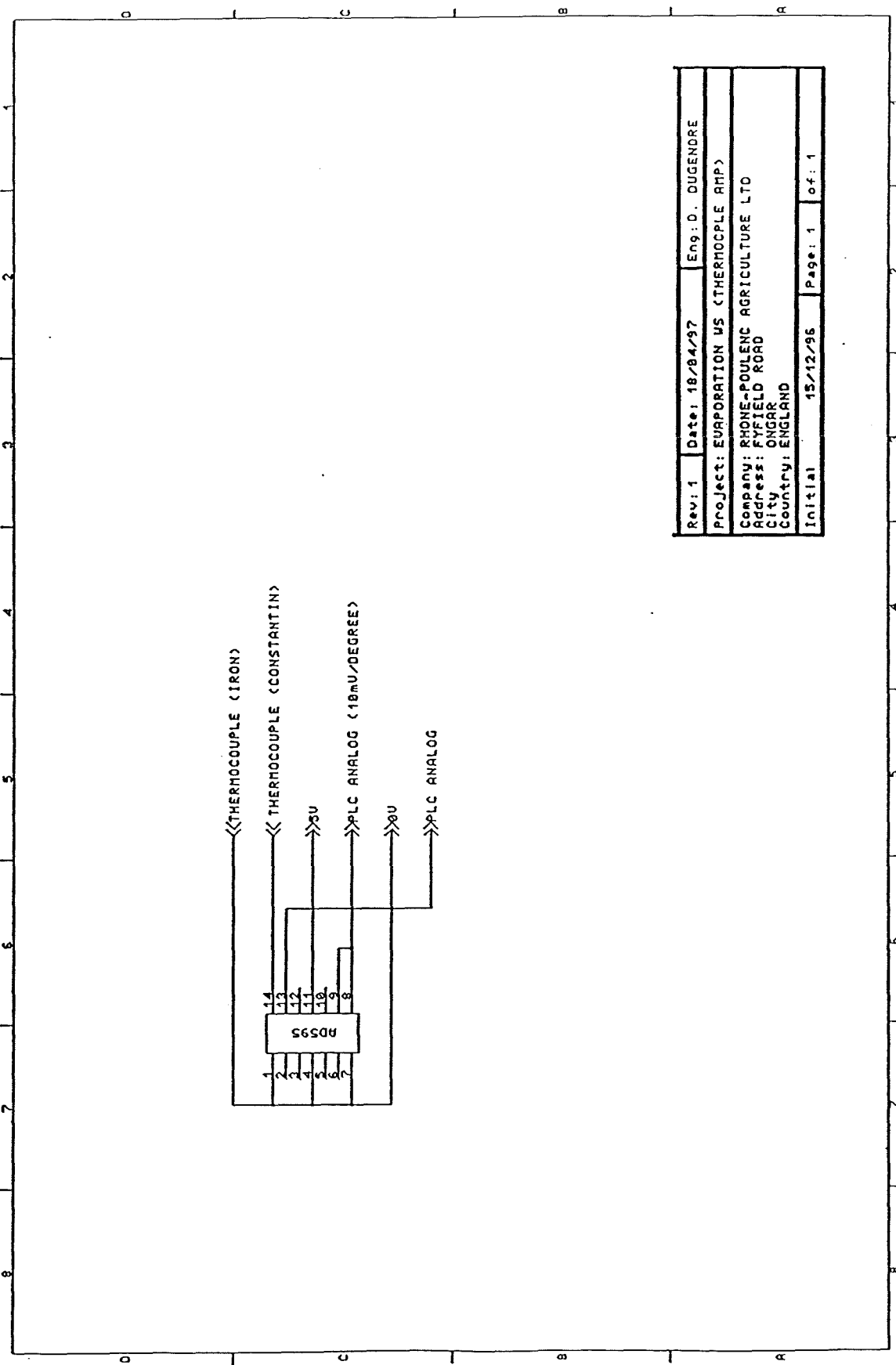
REFER TO DRAWINGS MA02, MA03 & MA04 FOR SENSORS, MICROSWITCHES AND SOLENOID WIRING.

TITLE		PNEUMATIC DIAGRAM		PLANT EXTRACTION WORKSTATION		DIMENSIONS IN MILLIMETRES (mm) TOLERANCES UNLESS OTHERWISE STATED +/- 0.1mm		REV		DATE		CHANGE	
								MATERIAL FINISH		DRAWN: D DUGENDRE		19 JANUARY 98	
								SCALE		Do not scale		FILE: G:\DATA\WORKSTAT\WAGERATR\PNEUM.DWG	

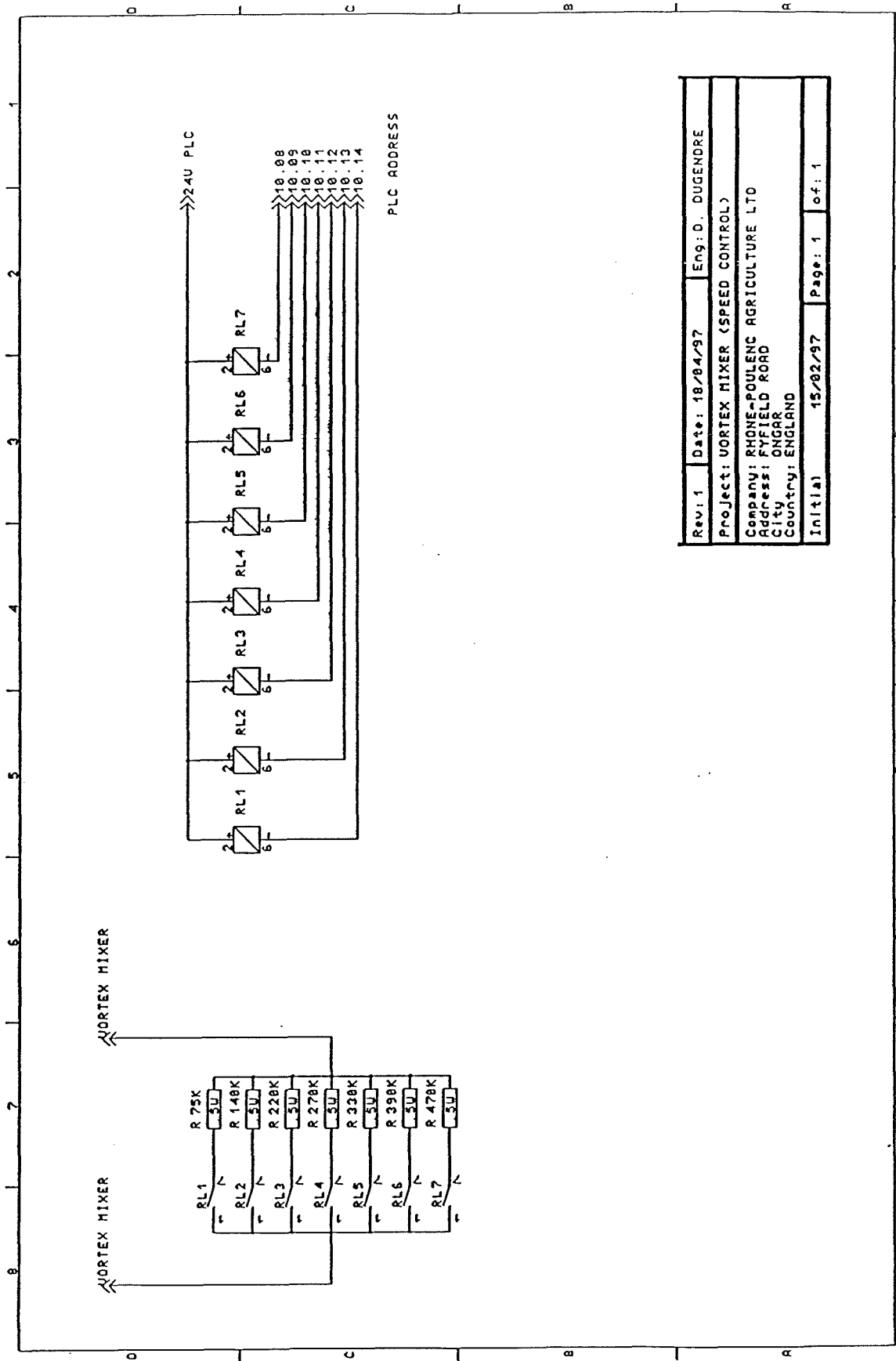
GPC Electrics Block Diagram



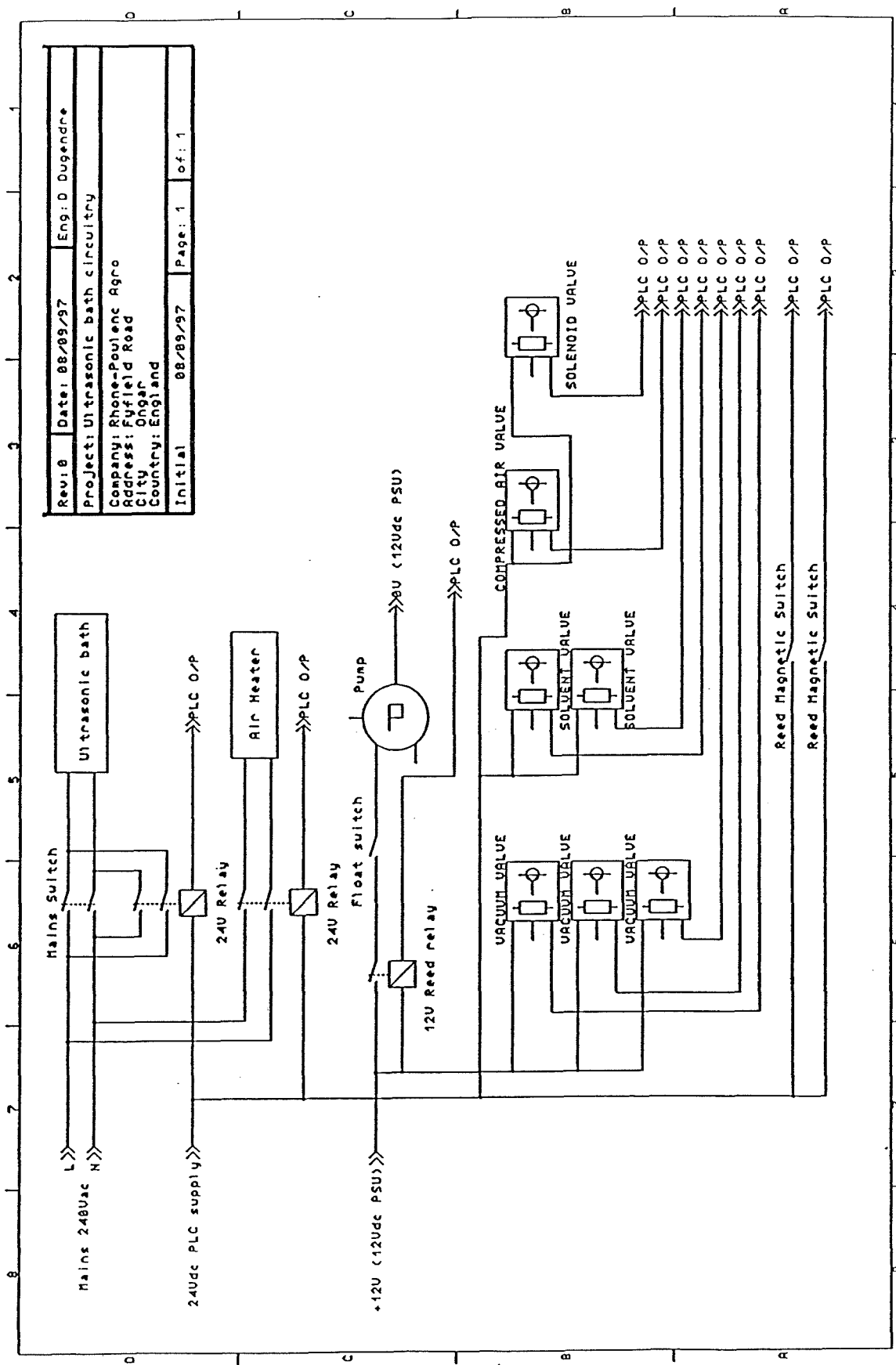




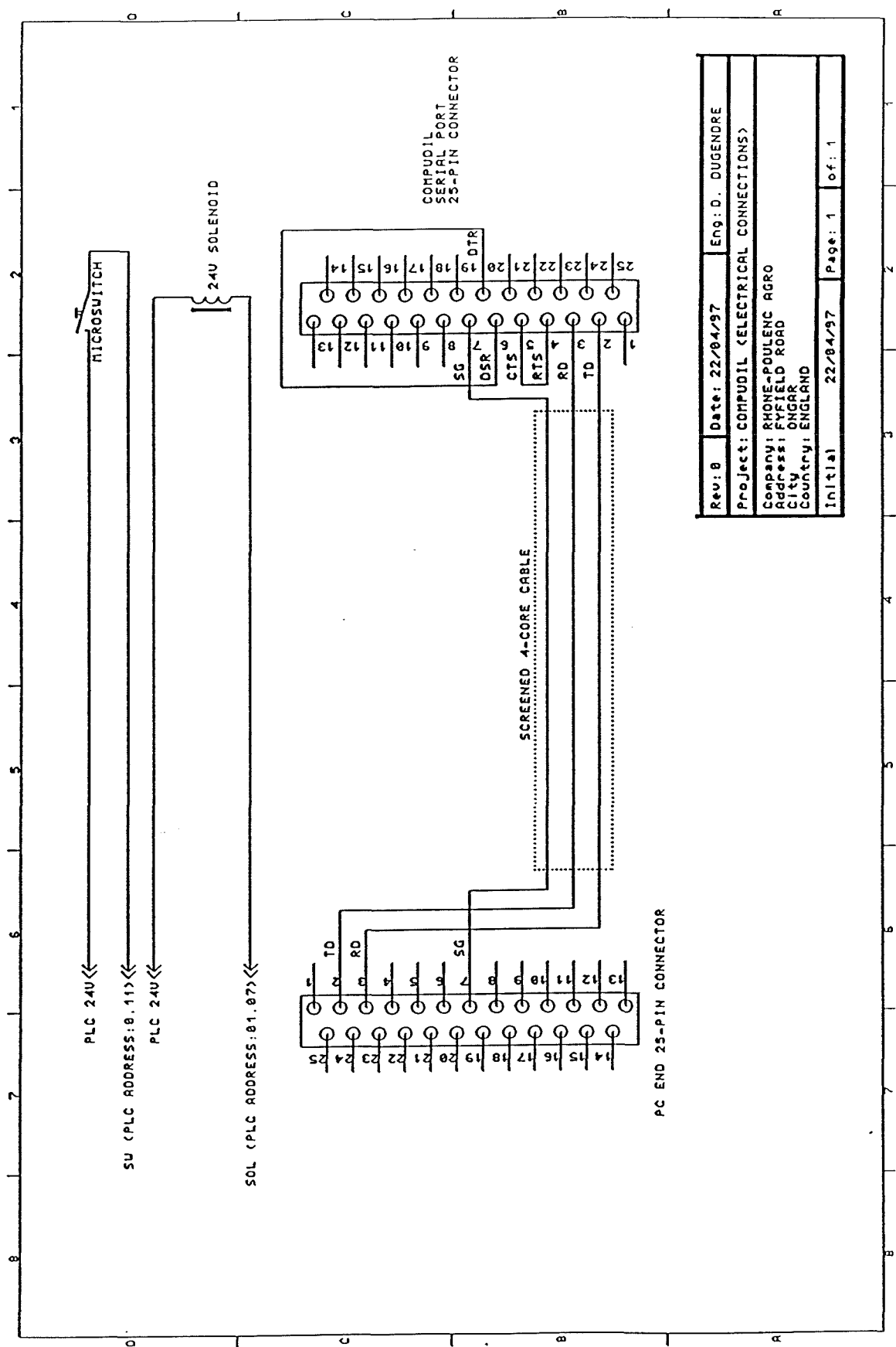
Rev: 1	Date: 18/04/97	Eng: D. DUGENDRE
Project: EVAPORATION US (THERMOCUPLE AMP)		
Company: RHONE-POULENC AGRICULTURE LTD		
Address: FYFIELD ROAD		
City: ONGAR		
Country: ENGLAND		
Initial	15/12/96	Page: 1 of: 1



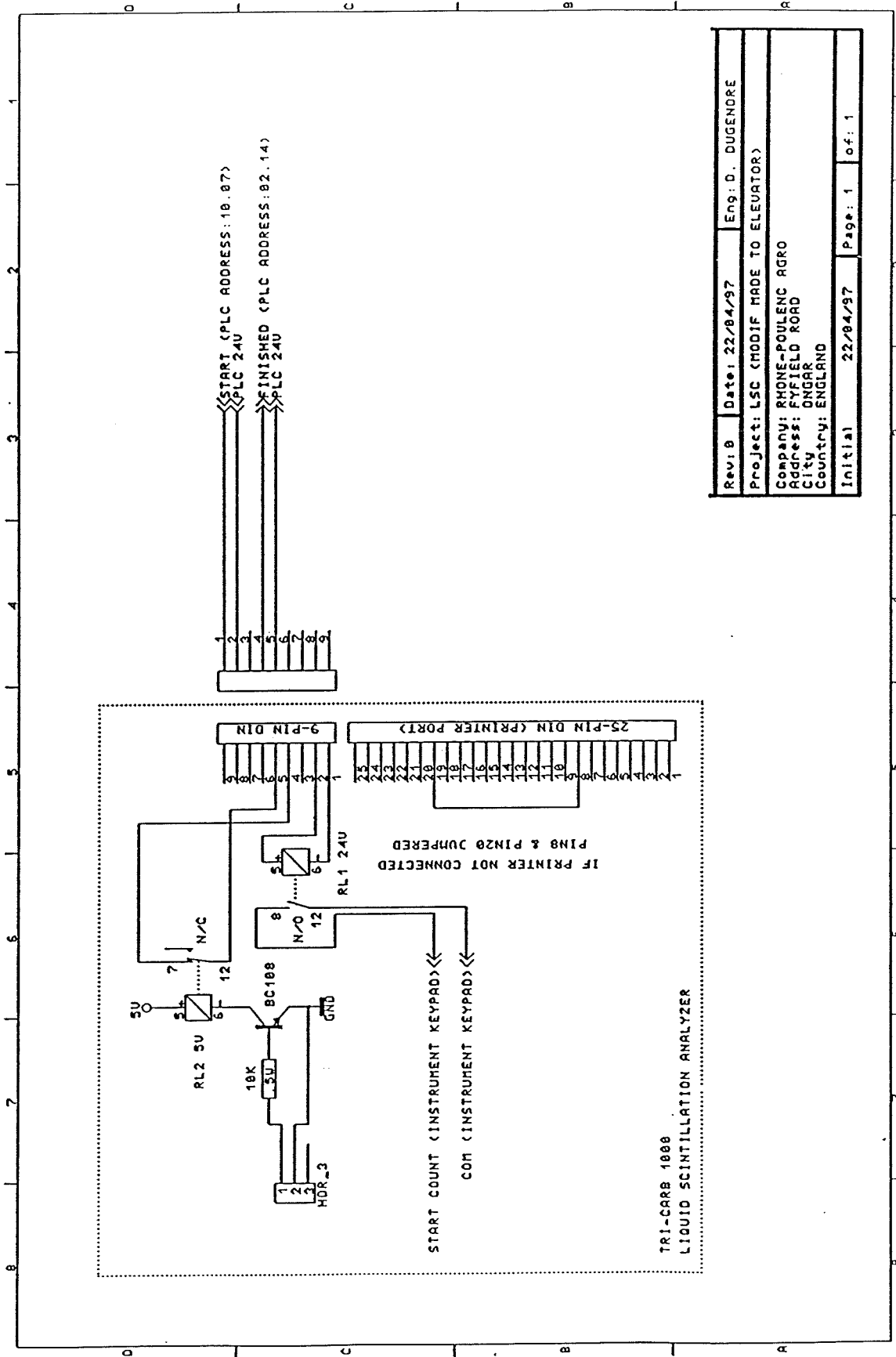
Rev: 1	Date: 18/04/97	Eng: D. DUGENDRE
Project: VORTEX MIXER (SPEED CONTROL)		
Company: RHONE-POULENC AGRICULTURE LTO		
Address: FIFEILD ROAD		
City: ONGAR		
Country: ENGLAND		
Initial	15/02/97	Page: 1 of: 1



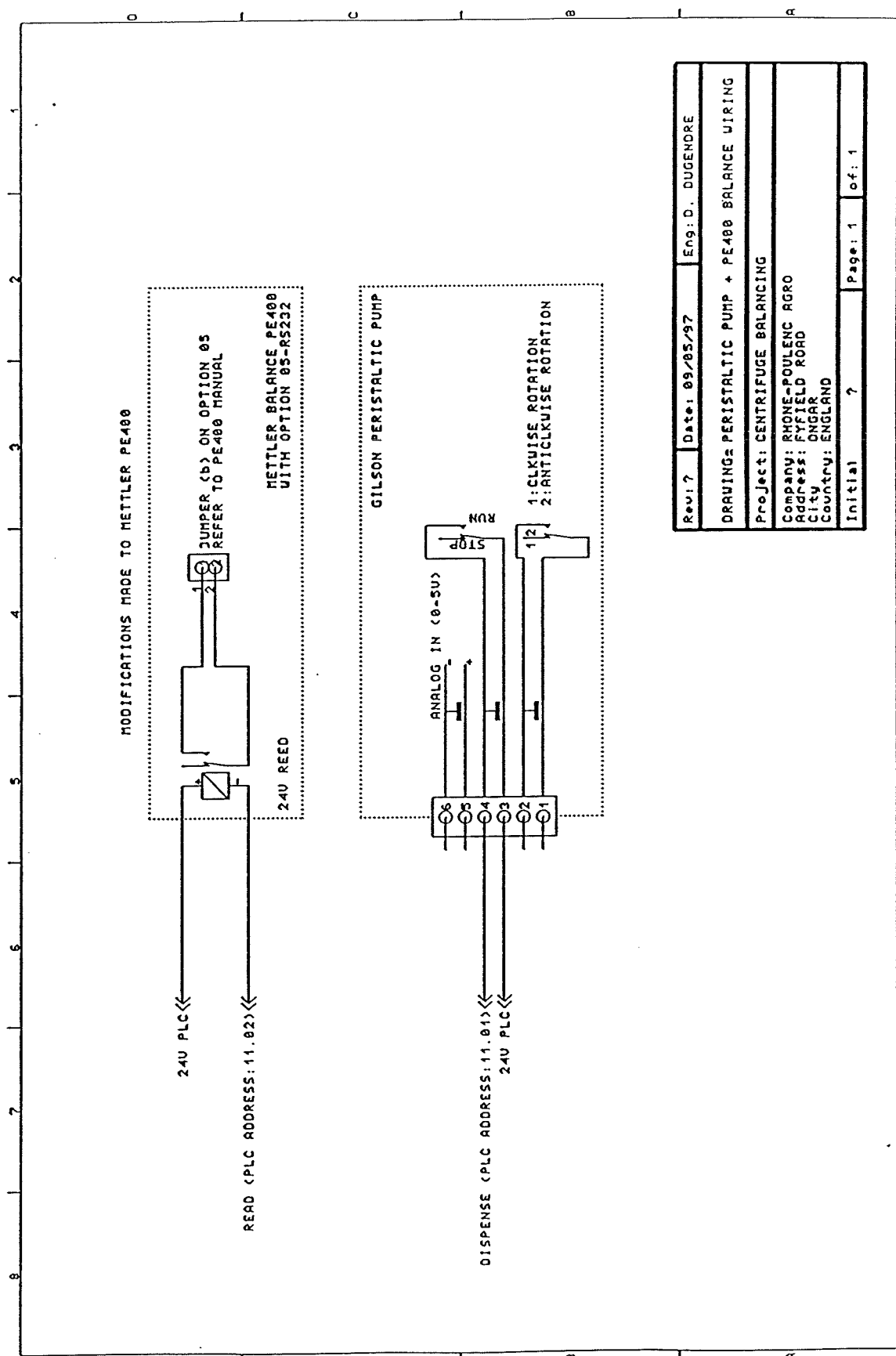
Revis B	Date: 08/09/97	Eng: D Dugendre
Project: Ultrasonic bath circuitry		
Company: Rhone-Poulenc Agro		
Address: Fyfield Road		
City: Onger		
Country: England		
Initial	08/09/97	Page: 1 of: 1



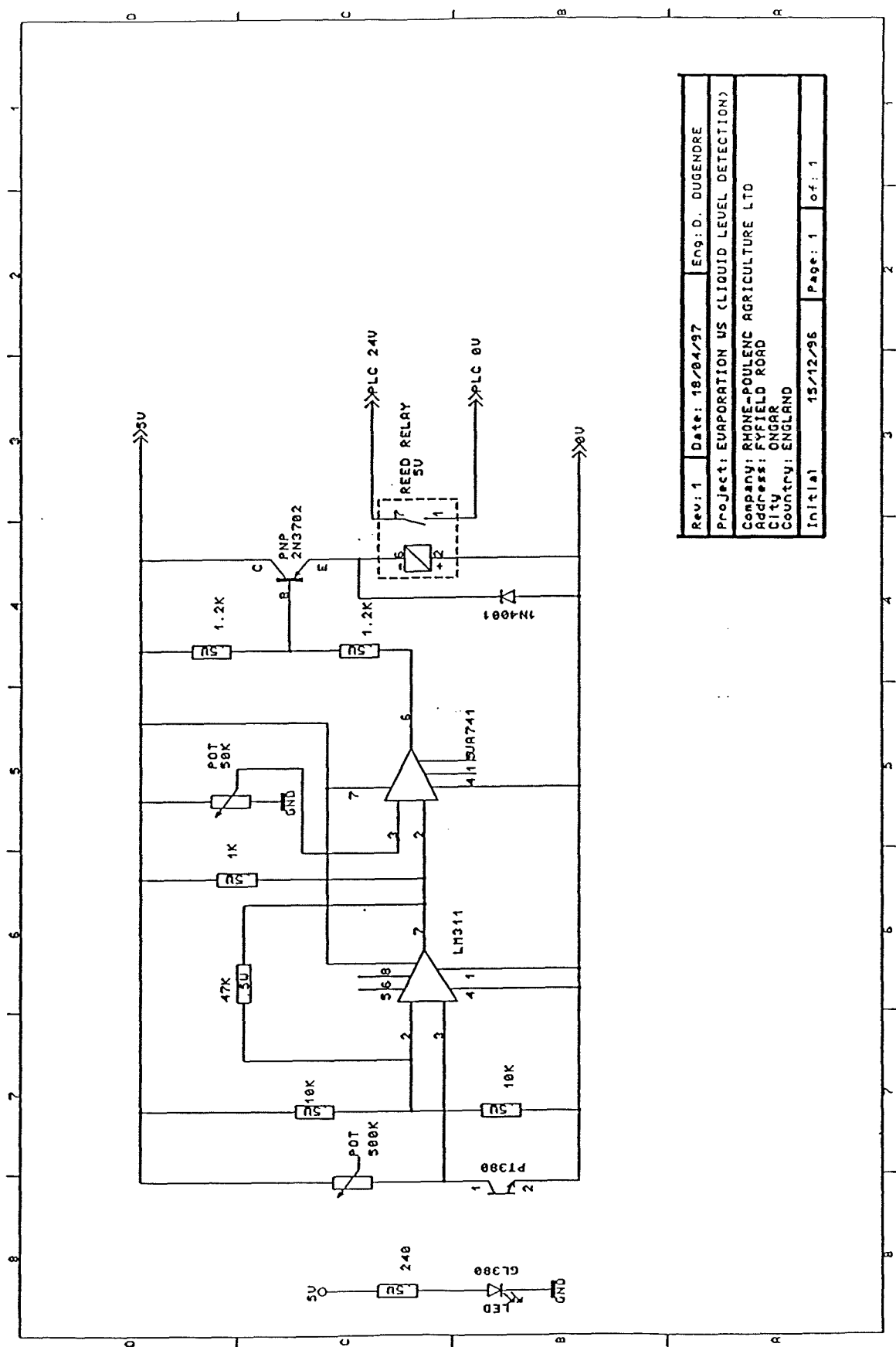
Rev: 8	Date: 22/04/97	Eng: D. DUGENDRE
Project: COMPUTIL <ELECTRICAL CONNECTIONS>		
Company: RHONE-POULENC AGRO		
Address: FYFIELD ROAD		
City: ONGAR		
Country: ENGLAND		
Initial	22/04/97	Page: 1 of: 1



Revi B	Date: 22/04/97	Eng: D. DUGENDRE
Project: LSC (MODIF MADE TO ELEVATOR)		
Company: RHONE-POULENC AGRO		
Address: FYFIELD ROAD		
City: ONGAR		
Country: ENGLAND		
Initial	22/04/97	Page: 1 of: 1

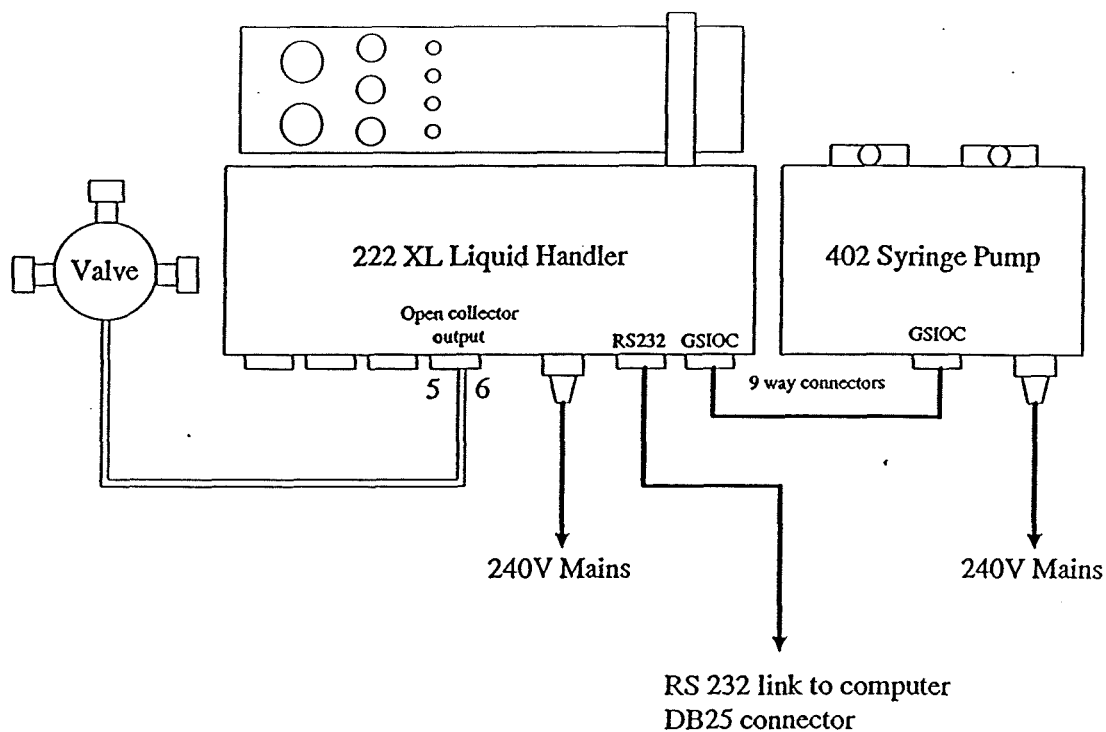




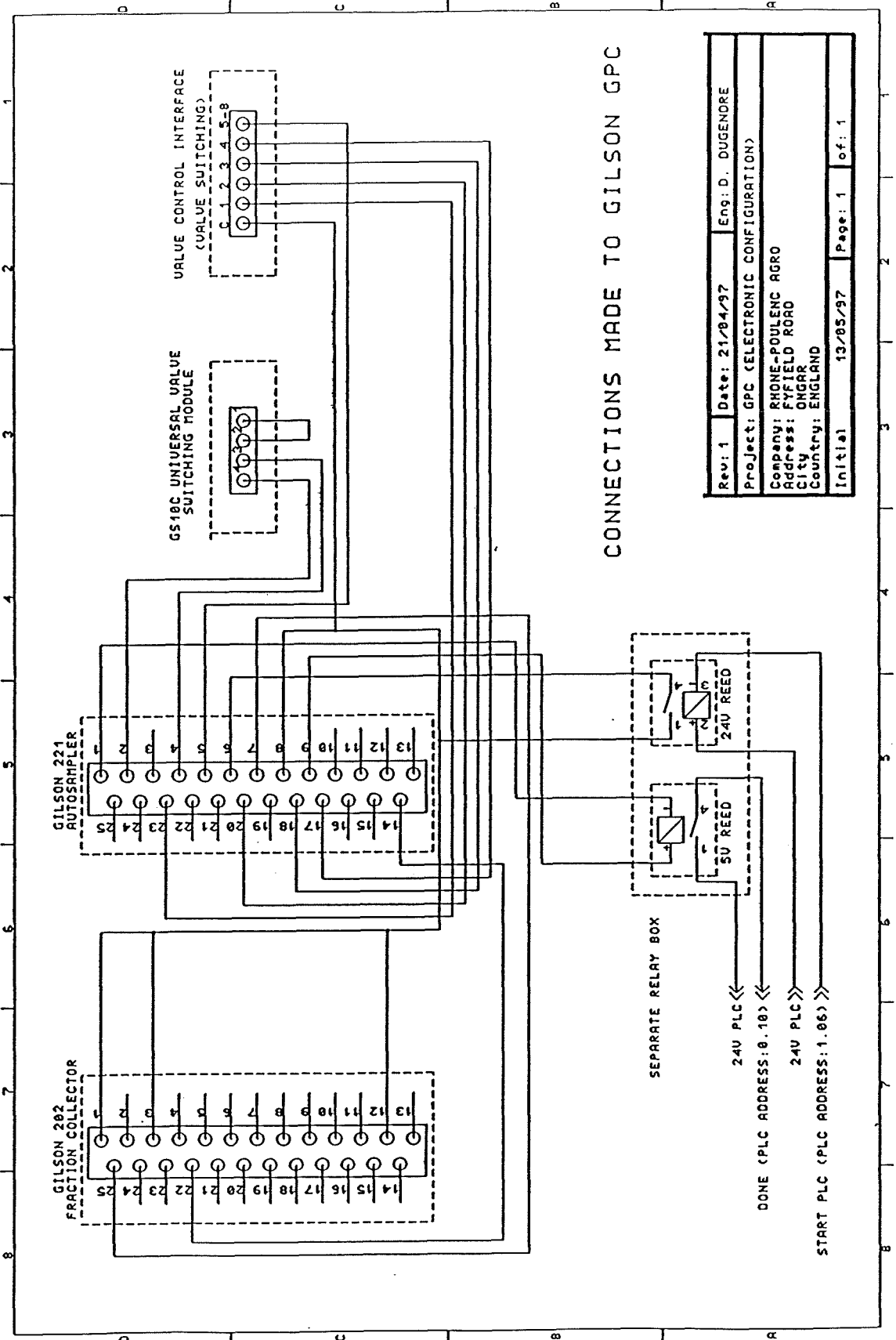


Rev: 1	Date: 18/04/97	Eng: D. DUGENDRE
Project: EVAPORATION US (LIQUID LEVEL DETECTION)		
Company: RHONE-POULENC AGRICULTURE LTD		
Address: EYFIELD ROAD		
City: ONGAR		
Country: ENGLAND		
Initial	15/12/96	Page: 1 of: 1



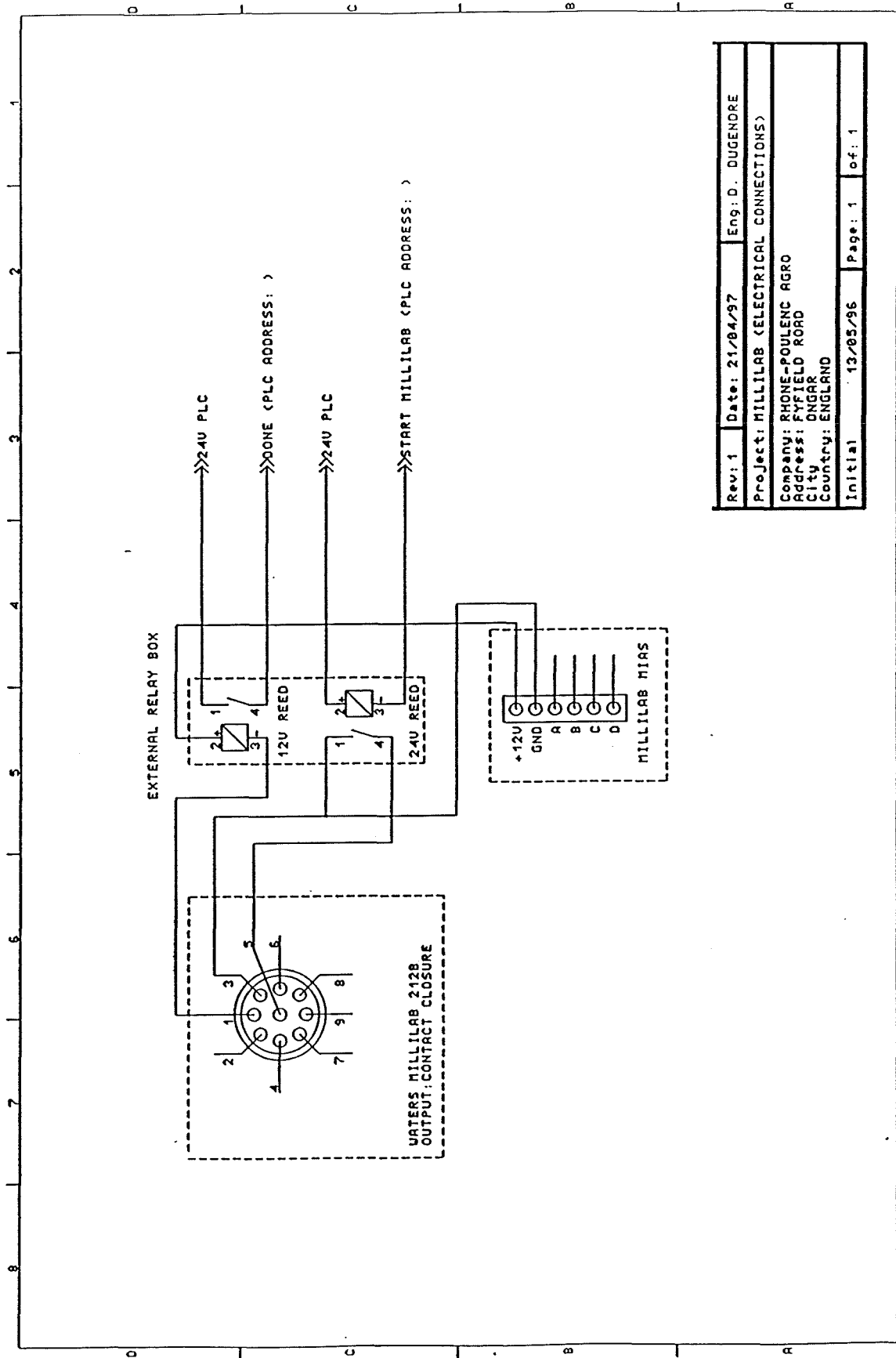


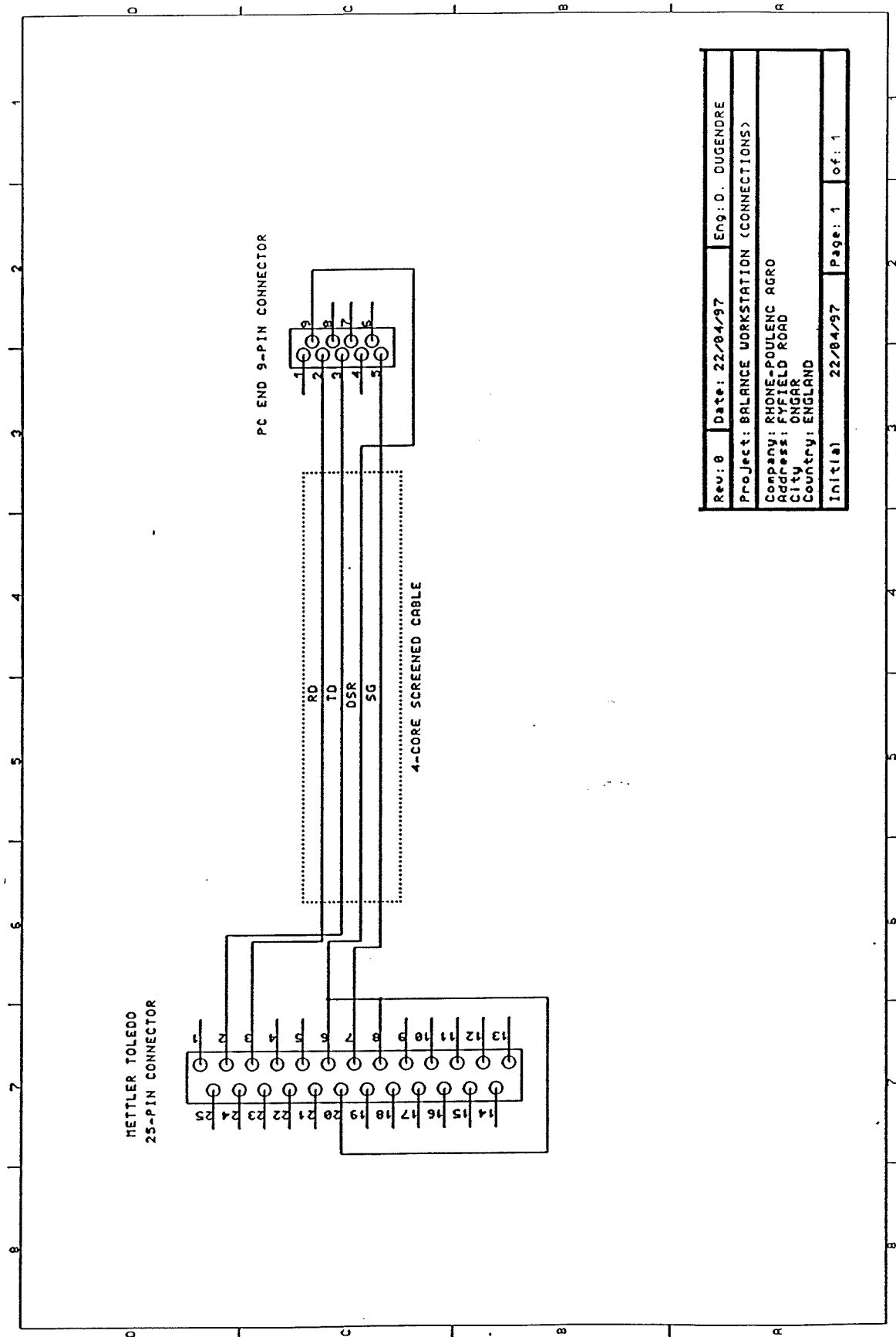
<b>Date:</b>	27/08/97	<b>Page:</b> 1/1
<b>Engineer:</b>	D Dugendre	
<b>Company:</b>	Rhône-Poulenc Agriculture Ltd	
<b>Address:</b>	Fyfield Road Ongar Essex CM5 OHW	
<b>Title:</b>	Pipette connexion	
<b>Project:</b>	ACAP P92072	



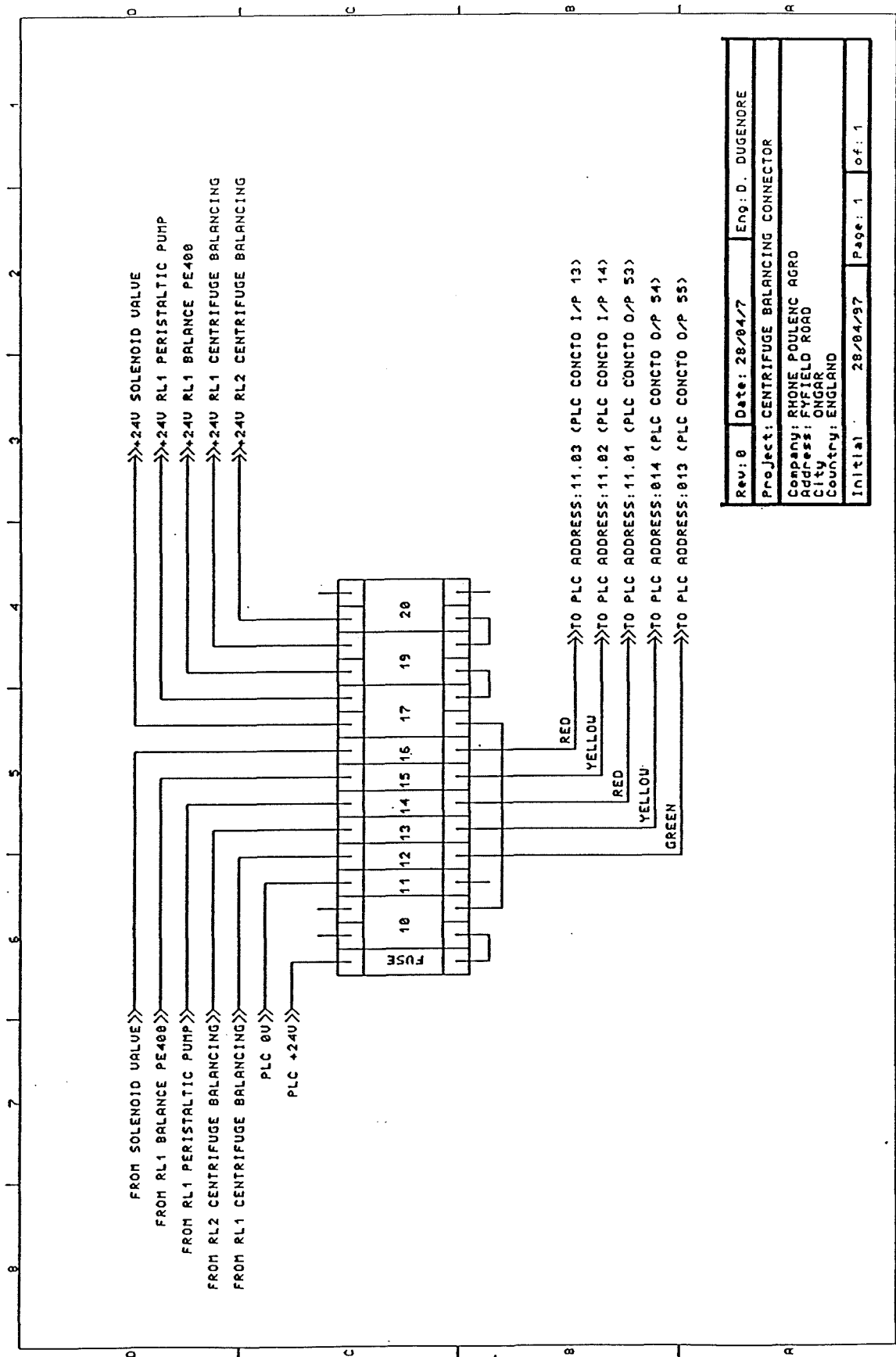
CONNECTIONS MADE TO GILSON GPC

Rev: 1	Date: 21/04/97	Eng: D. DUGENDRE
Project: GPC (ELECTRONIC CONFIGURATION)		
Company: RHONE-POULENC AGRO		
Address: FYFIELD ROAD		
City: OMSART		
Country: ENGLAND		
Initial	13/05/97	Page: 1 of: 1





Rev: 0	Date: 22/04/97	Eng: D. DUGENDRE
Project: BALANCE WORKSTATION (CONNECTIONS)		
Company: RHONE-POULENC AGRO		
Address: FYFIELD ROAD		
City: ONGAR		
Country: ENGLAND		
Initial	22/04/97	Page: 1 of: 1



Rev: 0	Date: 28/04/97	Eng: D. DUGENDRE
Project: CENTRIFUGE BALANCING CONNECTOR		
Company: RHONE POULENC AGRO		
Address: FYFIELD ROAD		
City: ONGAR		
Country: ENGLAND		
Initial:	28/04/97	Page: 1 of: 1