

## Transitioning analytical instrumentation from the laboratory to harsh environments\*

R. B. Turner

*Smiths Detection, 459 Park Avenue, Bushey, Watford, Hertfordshire, WD23 2BW, UK*

*Abstract:* The development of sensitive analytical instrumentation for autonomous operation in harsh environments is a difficult and expensive process. The management of risk in this process is aided by the use of technology readiness levels (TRLs) and awareness that the exponential growth of costs through development militates very strongly against progressing the development prematurely. It is also essential to have a very clear view of the intended outcome at the earliest possible stage and to test the systems in realistic environments as soon and as often as possible. Economic production of large quantities of instruments requires considerable attention to the details of the production process and the control of materials; there is a constant battle against contamination. Future instrumentation might involve a combination of analytical techniques, but the desire for more selectivity and sensitivity must be balanced against cost, complexity, and reliability.

### INTRODUCTION

For nearly 20 years, my company has been involved with the design and development of instruments employed in the field to detect traces of chemicals including illicit drugs, explosives, and chemical warfare agents. These instruments, mainly based on ion mobility spectrometry (IMS) [1,2], include the chemical agent monitor (CAM) shown in Fig. 1, the automatic chemical agent alarm (GID-3 ACADA) shown in Fig. 2, and the volatile organics analyzer (VOA) [3], which is currently in operation on the International Space Station.



**Fig. 1** Chemical agent monitor (CAM).

---

\*Lecture presented at the IUPAC Workshop, Impact of Scientific Developments on the Chemical Weapons Convention, Bergen, Norway, 30 June–3 July 2002. Other presentations are published in this issue, pp. 2229–2322.



**Fig. 2** GID-3 ACADA.

We generally hope to manufacture thousands of each instrument—over 50 000 CAMs have been made—although the VOA is an exception for obvious reasons. Our products are sensitive (generally in the region of parts per billion or lower), rugged, and automatic (they do not require the operator to interpret the results). In this article, I describe some aspects of the process by which technology is taken from the laboratory to use in demanding environments—and some of the particular problems that have been encountered in the manufacture of trace detectors.

## PRODUCT DEVELOPMENT

The development of a new product requires investment. In order to justify this investment, a number of questions should be asked and answered before the process can start. These questions include:

- What are the technological risks in the proposed development?
- What are the estimated costs of the proposed development?
- What is the proposed time scale?
- What is the prospective market—does this justify the investment?

The management of risks in high technology development has become an area of focus for governments and industry—largely as a result of bitter experience.

## TECHNOLOGY READINESS

One way of looking at the risks involved in the development of technology into products was developed by NASA in the late 1990s and has subsequently been employed by both the U.S. [4] and U.K. governments.

In this methodology, there are nine TRLs defined briefly as follows:

1. Basic principles of technology observed and reported
2. Concept and/or application formulated
3. Analytical and laboratory studies to validate analytical predictions
4. Component and/or basic subsystem technology validated in lab environment
5. Component and/or basic subsystem technology validated in relevant environment
6. System/subsystem technology model or prototype demonstrated in relevant environment
7. System technology prototype demonstrated in an operational environment

8. System technology qualified through test and demonstration
9. System technology qualified through successful mission operations

The costs involved in the development of a new product rise exponentially as the program moves through the various stages. Conversely, the risks (both technical and in time scale) should fall as the program approaches completion. One of the objectives of the technology readiness approach is to ensure that an appropriate level of technological maturity has been reached before major investment is made. This does not generally mean that all the risks disappear before the end of the project, but they do change in nature. Early in the process, the scientific risks will predominate. Risks in the later stages will be mainly associated with engineering issues.

It is extremely important to have an accurate specification of the final system; this implies a very clear view of the purpose and modes of use. The specification must be complete, but not excessive. For example, if the equipment is required to be operational within 5 min after long storage then this must be explicitly stated in the specification—it is very difficult to prevent long-term build-up of contamination when trace detectors are stored. On the other hand, asking for high measurement accuracy when only qualitative results are really needed can lead to high cost for no purpose.

### **COSTS OF DEVELOPMENT**

The two most recent development programs that we have carried out were both essentially modifications of technology that had already been “proven” in previous products—corresponding to level 5 in the readiness scale. Each of these programs has cost approximately \$3 million and involved teams of about 20 people. The development teams include a mix of disciplines—scientists, mechanical engineers, electronics (analog and digital) engineers, production engineers, process engineers, software engineers, safety engineers, test engineers, and logistics experts, in addition to a planner and a project manager. The development time (from level 5 to level 9) is typically 1.5–2 years.

Our development programs involve a considerable amount of time and effort in testing; this starts to consume serious money at TRLs 5 and 6. The tests range from relatively straightforward environmental tests for ruggedness to operational tests in realistic environments. The latter should be carried out as soon as practical and as extensively as possible; we have had some unpleasant shocks as a consequence of a lack of knowledge of the chemical background.

### **WHERE DOES ALL THE MONEY GO?**

It is not necessarily the most scientifically challenging areas that take the most resources in the development process. One of the most difficult aspects of the design of portable instrumentation is packaging—how to get all the electronics, pumps, sensors, displays, and input/output interfaces in the most compact and ergonomic form. The outside case of the instrument can often be one of the most expensive items both to design and to purchase. The case has to provide electrical screening for both radio-frequency emissions from the instrument and to prevent external fields from affecting its operation. It has to be sealed against the elements, and it must be easy to assemble. It has to be as light as possible, and yet it must be robust.

### **DESIGNING FOR PRODUCTION**

Trained and experienced people can assemble a small number of instruments using experience and knowledge to tackle any problems that arise. However, this approach becomes less and less viable as the production rate rises and larger numbers of people must be involved. Documentation both of the product and the process becomes vital, and considerable effort can be expended in establishing processes that are robust and a design that can be reliably reproduced with the minimum of specialist

knowledge or “black art”. At TRLs 6 and 7, the investment in production tooling and process engineering becomes significant.

### Designing the membrane

Whereas it is desirable to control or specify all aspects of manufacture, sometimes this is not achievable. For example, a simple but vital component in some of our instruments is a thin (12  $\mu\text{m}$ ) silicone rubber membrane (shown in Fig. 3 mounted on a holder).

This membrane acts as the inlet to the system; sample vapors dissolve in it and permeate through to the analyzer within the instrument. The membrane provides selectivity (particularly against water vapor), and it prevents contamination from dust.

Each membrane was individually tested (this of course is a waste—we would prefer that they be 100 % leak-free), and a high and variable failure rate was observed. Test results varied from 90 % pass to 90 % fail on a batch-to-batch basis. The problem was tackled systematically; initially the processes within the company were refined to ensure that the handling, assembly and test were not contributory factors. The failure mechanism was analyzed and determined to be primarily a result of particle inclusion—a 10- $\mu\text{m}$  particle in a 12- $\mu\text{m}$  membrane is clearly going to lead to problems. Next, the subcontractors who mold the item were involved, and clean-room processes and environments were instigated. The net result was a reduction in failure rate—but we still had failures. Discussions with the supplier of the bulk silicone revealed that whereas the material is made in a relatively clean area, it is not a filtered air facility. They were unwilling to install an expensive plant and implement cost-raising procedures to satisfy a customer who purchased less than 0.01 % of their output.

We still live with testing (and failing) membranes.



Fig. 3 The membrane.

### Contamination in trace detector manufacture

For trace detection equipment, ensuring adequate cleaning of components used in the sensor assembly becomes a key issue. A significant proportion of the space on our production floor is occupied by washing and baking machinery. Contamination of the air supplies that are used to purge the ovens can stop the production process, so these are continuously monitored and a back-up supply is available.

The production process sometimes appears to be a continuous fight against contamination. We have had problems with pollution from traffic and from solvent emitted from a factory next door—although these are well below permitted levels. We have to control the polish on the factory floors and production operators' perfumes and deodorants.

### Designing the pump

We designed a miniature diaphragm pump (Fig. 4) to circulate air within one of our instruments. In order to keep the costs reasonable, it is constructed of a number of molded plastic components.

After a great deal of testing and experimentation, a particular grade of polypropylene was chosen as providing the right combination of chemical cleanliness (after a washing and baking process) and mechanical properties. The production of large quantities commenced, and all went well for a year or so. Then it was noticed during the test of completed instruments that contamination had started to appear. Every polypropylene component in the factory was found to be grossly contaminated. The investigating team was spurred on by a production line with no output and various offers of help from senior management. They eventually found that the supplier of the polypropylene molding granules had closed down the plant that supplied the original material and had moved production to a new plant in a different country. This new plant used a different antioxidant in the formulation, so that whereas the product met the manufacturer's specification it did not meet our more stringent requirements. Current procedures call for batch testing of molded components to detect such problems at an early stage.

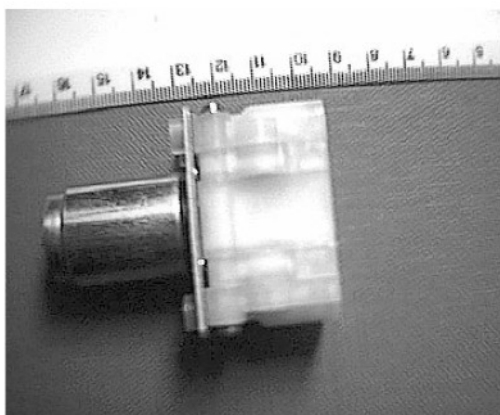


Fig. 4 The pump.

### THOUGHTS ON FUTURE INSTRUMENTATION

There is a continual demand for instruments of greater sensitivity. As sensitivity levels are pushed higher, more problems are encountered with the chemical background (these problems might be most severe in industrial facilities). More selectivity must go hand-in-hand with greater sensitivity. Approaches for higher selectivity include the use of a separation stage such as gas chromatography. However, this gives penalties in the form of increased complexity, longer analysis times, and problems with the provision of inert carrier gas.

Adding chromatography to a detection technique that shows extreme sensitivity toward specific groups of materials can produce a system that combines both selectivity and sensitivity. One example of this is the chemiluminescent method for the detection of explosives. This is an excellent method for explosives that contain nitrogen. However, increased use by terrorists of explosives based on peroxides makes the method redundant. To make a technique future-proof, it must be capable of responding to as wide a range of compounds as possible, which places even more demands on selectivity.

Combining techniques that rely on different principles for their operation is an attractive approach for trace detection. Examples might be combinations of IMS and cavity ring-down spectrometers [5]. These techniques are extremely sensitive and sufficiently small to make a combination possible in a portable instrument. Target materials might be more reliably confirmed by detection by multiple tech-

niques. Unfortunately, the fact that the methods measure different parameters is not a guarantee that they will together give more discrimination. In fact, one might take the opposite view based on the fact that they all respond to the target set of materials.

Cost and reliability would both be adversely affected by the inevitable increase in complexity inherent in the use of multiple analytical techniques. Whether any gain in performance is balanced by these adverse factors will probably not be known until such a project is attempted.

## CONCLUSION

Designing and manufacturing sensitive portable instrumentation is an expensive, complex, and risky activity. Before undertaking such a task, it is essential to have a very clear view of the desired objective. At the earliest possible stage, the specification should be detailed, and its practicality should be determined by testing in a realistic environment. Major effort should be devoted to the management and amelioration of risk.

## REFERENCES

1. G. A. Eiceman and Z. Karpas. *Ion Mobility Spectrometry*, CRC Press, Boca Raton, FL (1994).
2. R. B. Turner and J. L. Brokenshire. *Trends Anal. Chem.* **13** (7), 275–280 (1994).
3. P. G. Bass. *Spaceflight* **42**, 139–144 (2000).
4. DoD 5000.2-R, Mandatory Procedures for Major Defense Acquisition Programs (MDAPs) and Major Automated Information System (MAIS) Acquisition Programs.
5. R. J. Saykally and R. Casaes. *Laser Focus World* **37**, 159–162 (2001).