The Role of Analytical Chemistry in Industry & Environment

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INTRODUCTION

The properties of every substance, from the air we breathe to the vast range of materials and products which we use in our business and private lives are directly or indirectly a function of chemical composition. These properties are influenced for better or worse, by the presence or absence of one or more chemical species, sometimes at concentration levels which are barely detectable. An adverse effect on properties may, in some situations, be a matter of inconvenience to the user of a product or material; in other circumstances however the consequences may be more serious, resulting in injury or even loss of life, not merely to the user but to those around him. Consider the consequences of the failure in flight of a vital component in an aircraft, the contamination of a foodstuff with a toxic metal or compound, or the presence of even a few milligrams of asbestos per cubic meter in the air we breathe.

Chemical composition however is not just associated with risk of injury. The commercial value of virtually all materials and many products is related to their content of intrinsically valuable substances (e.g. a precious metal) or to their suitability for the purpose for which they are intended and this almost invariably is a function of how closely they adhere to a compositional specification.

It is obvious therefore that chemical composition in many instances decides the value of products and materials which form the basis of national and international trade. As a consequence, it is not surprising that there are more government regulations, standard specifications and trading agreements associated with chemical composition than all other properties put together. They would have little meaning however if composition could not be established conveniently and to a degree of reliability satisfactory to all the parties concerned. It is here that analytical chemistry plays its role.

WHAT IS ANALYTICAL CHEMISTRY?

It is a well established scientific principle that one should, when discussing a subject, define ones terms and 'analytical chemistry' should not be treated any differently. There is a slight problem however because, despite the tens of thousands of persons throughout the world who would claim to be analytical chemists, the profession as a whole has shown a marked reluctance to define itself, principally because it is felt that any formal definition would be either too restrictive or too broad to be meaningful. A definition which, it is suggested, would seem to describe the major part of the spectrum of activities which are covered by the term analytical chemistry could be written as follows:

'*Analytical chemistry is the science concerned with the systematic identification or characterisation of established chemical species and their determination to known degrees of certainty at any level of concentration and in any matrix in which they may occur*'.

Two points in the definition merit comment. Firstly, the analytical chemist is concerned with established chemical species; it is not his function to determine the structure of hitherto unknown compounds and secondly, the scope of analytical chemistry extends well beyond establishing composition on an elemental basis. In practice, the analytical chemist must differentiate between elements in free and ionic form, between valence states, between complexed and uncomplexed metals, between stereoisomers and between phases in complex mixtures.

Table 1 gives some examples of the latter point and also illustrates the importance of being able to distinguish between the different forms in which elements can be found.

TABLE 1	IMPORTANCE OF CHEMICAL SPECIES PRESENT AS DISTINCT FROM THE ELEMENTAL COMPOSITION
ELEMENT	**POSSIBLE FORMS**
Si	SiO_2*, CH_3 CH_3 / Si -- O -- Si -- O -- / CH_3 CH_3
Sn	Sn^{+2}, Sn^{+4}, $Sn(Me)_2$*, $Sn(Me)_3$*
Cr	Cr^{+3}, Cr^{+6}*
F	HF*, CaF_2
Cl	Cl^-, Cl_2*

* most toxic form

THE DEVELOPMENT OF ANALYTICAL CHEMISTRY

Analytical chemistry has been practiced in crude form from ancient times but began to develop as an objective exercise during the 17th century. Table 2 lists some of the milestones in its development. For those interested in historical aspects of the subject, references 1 – 4 are a selection of the literature on the history of analytical chemistry.

Throughout the 17th, 18th, and 19th centuries, analytical chemistry was largely empirical, relying on experimental data which was not explained by theory. It was only from the beginning of this century however that theories of solution chemistry, atomic structure, spectroscopy, etc. began to offer theoretical explanations for established analytical procedures and show the way for the development of others. Over the centuries, analytical chemistry in fact developed alongside chemistry as a whole and there was little distinction between the two. During this period Irish chemists played their part and some of the more important names are given in Table 3. For more information on the Irish connection, two excellent reviews have been published (5, 6).

In this century, analytical chemistry, like most other areas of science and technology, has expanded at a rate that would have been incomprehensible to previous generations. For the most part the theory of present day techniques developed ahead of the hardware. For example, the principle of atomic absorption was known around the turn of the century but had to await the development of the hollow cathode lamp and photomultiplier tubes to become a practical proposition. Likewise, an Ag/AgCl ion selective electrode was described in 1937 but it was not until 1966 that one was built using silicone polymers which were not available in 1937.

The development of analytical chemistry in terms of output of analytical data has increased almost exponentially since the war. This is largely due to increasing demand by a more informed society for more stringent control in the areas such as the environment, health and safety, consumer protection etc. and the increased sophistication of the materials and manufacturing techniques used in industry. This demand has led to a large increase in the amount of research being carried out in analytical chemistry; in fact this area has the most rapidly developing output of research literature of all the sciences, including chemistry as a whole. This is demonstrated by the data on 'doubling time' for scientific literature which is given in table 4. In keeping with other areas of technology, the output of analytical data has been greatly facilitated by the automation made possible by the developments in the area of microelectronics.

TABLE 2 SOME MILESTONES IN ANALYTICAL CHEMISTRY

1650	- Use of H_2S
	- Study of acid base indicators

1750	- First titrations (gravimetric) - First use of graduated burette - First C, H analysis by combustion - Detection of Cu by electrolysis (1800)
1800	- Start of emission spectroscopy - Improvement in C, H analysis - Dumas method for nitrogen - Hardness of water
1850	- Ag/Cl titration improved - Methyl orange and phenolphthalein - Kjeldahl method for nitrogen - Emission spectroscopy firmly established - Beer-Lambert law - Nernst equation - Food and Drugs Acts 1860 and 1875 - appointment of public analysts
1900	- Theoretical basis of past and much of present day Analytical Chemistry established
1950	Big developments in instrumental methods of analysis including automation
1980	

EDUCATION IN ANALYTICAL CHEMISTRY

Universities and other educational establishments have responded to the need for persons trained in analytical chemistry by establishing formal facilities for teaching the subject. Today in almost every country in the world there are a number of universities with formal lectureships, chairs and even whole departments devoted to analytical chemistry. Table 5 shows a comparison of the position in 1980 with that in 1960 with regard to the teaching of analytical chemistry in universities in the United States. Of significance is the increase in the number of PhDs, both in absolute terms and as a percentage of the total.

In Northern Ireland there is a long established chair of analytical chemistry at Queen's University Belfast but in the Republic universities were slow to respond to the increasing need for training in the subject. Recently however a lectureship in analytical chemistry was established in University College Cork and M.Sc. and PhD course will soon commence there. It should be pointed out however that, in contrast to the universities, the technological colleges particularly the Regional Technical Colleges, responded actively to the need for training in analytical chemistry at technician level.

In Europe the situation varies from one country to another but in all there are facilities for the teaching of analytical chemistry and research in universities and colleges of technology. For those interested in the situation in Europe, the proceedings of a special session of education in analytical chemistry held during Euroanalysis III in Dublin in 1978 provides a comprehensive review (8).

TABLE 3 SOME IRISH ANALYTICAL CHEMISTS

<u>Robert Boyle (1627-1691)</u>
First to use the term 'chemical analysis'
First to use H2S in analysis
First to study plant extracts and to use cuprammine ion as acid/base indicators

<u>Richard Kirwan (1733-1812)</u>
Used ferrocyanide to determine iron
Published book on the analysis of mineral waters

<u>James Emerson Reynolds (1844-1920)</u>
Prof. of analytical chemistry at RDS
Published on the analysis of phosphate fertilizers and on the valuation of manganese ores
First to introduce quantitative analysis into Irish training course for chemists

<u>Charles A.Cameron (1830-1921)</u>
First public analyst in Ireland – third in British Empire
Published on the analysis of milk water and food.

TABLE 4 DOUBLING TIME FOR TECHNICAL LITERATURE (7)

	Years
Analytical Chemistry	13.9

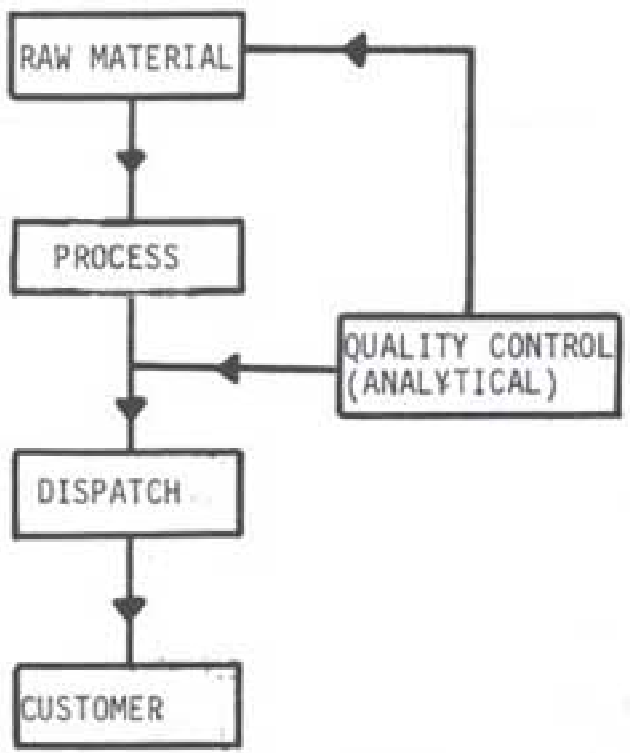
Chemistry Total	14.5
Biology	16
Physics	19
Electrical Eng.	20
Psychology	25

ROLE OF ANALYTICAL CHEMISTRY IN INDUSTRY

A simplified flow chart for any industrial process would be as shown in Figure 1. This represents three basic stages – the intake of raw material, its conversion to product in whatever process is involved and the dispatch of the product to the customer. The role of the analytical chemist in the quality control function with regard to raw material and finished product is well known but in fact it goes well beyond this.

A more realistic picture of an industrial process is given in Figure 2 which shows the many areas other than simple quality control in which analytical chemists are involved. This involvement need not necessarily be solely by in-house analytical chemists; in some cases, persons external to the organisation will have a voluntary or statutory role to play in the overall process. For example, the 1955 Factories Act (9) and the many regulations made under it authorises inspectors from the Department of Labour to examine certain industries which use or produce toxic or dangerous materials such as asbestos, silica dusts, benzene, lead fumes, etc. The inspection normally includes the taking and analysis of samples of factory atmospheres, materials in use and even the monitoring of the level of toxic substances in the body fluids of workers. Similarly, not only the company’s own analytical chemists but those from external agencies such as the Departments of the Environment and Health, An Foras Forbatha and the IIRS may be involved in monitoring the disposal of gaseous liquid and solid waste created in the process.

FIGURE 1 SIMPLFIED FLOW-CHART OF AN INDUSTRIAL PROCESS



The increasing cost of raw materials has led to an increased amount of recycling and it will be the responsibility of the analytical chemist to ensure that the quality of the recycled waste is suitable for reprocessing.

Packaging is an extremely important part of any industrial process and in recent years has developed into an industry in its own right. Packaging materials serve a number of functions the most important of which are to preserve product quality for a specified period – the 'shelf life', to protect the product from physical damage while in transit or on display and to increase customer appeal. Some of these functions may be in conflict. For example, with certain foodstuffs, brightly printed wrappers may attract potential customers but unevaporated ink solvent residues can taint the product. Some food packaging materials are controlled by law to ensure that they do not transfer toxic substances to the foodstuff. A recent example of this is an EEC Directive which limits the level of vinyl chloride monomer which may be in Food

Packaging materials (10).

TABLE 5 ANALYTICAL CHEMISTRY IN U.S. UNIVERSITIES (21)	1960	1980
No. of PhD granting Departments	125	184
No. of 'analytical' faculty members	244	424
% of total faculty	10.4	10.8
PhDs per annum in analytical chemistry	55	182
% of total PhDs in chemistry	5	11.9

It is necessary therefore for packaging materials to be monitored for functional suitability and for safety reasons in certain instances.

It is not intended to discuss the analytical chemist's role in product development and trouble shooting in the plant itself but in each of these areas he has an important support function to other scientists and technologists.

An area in which the analytical chemist has an important, if often unrecognised involvement, is in the semi-commercial area of product specification: changes to meet customer needs and customer complaints involving no compliance with specification, unsuitability of the product for his needs, contamination of the product, etc. Disputes between customer and supplier may be resolved by negotiation but in many cases they can lead to litigation and involve 'referee' analyses by independent analytical laboratories. In these cases therefore analytical chemists may find themselves involved in all the intrigue of litigation and their results being instrumental in deciding the outcome of disputes involving thousands and even millions of pounds. A very high degree of professional and scientific integrity must be exercised in situations such as this.

Worker wearing a personal air sampler to monitor dust produced in grinding operations, using a battery-operated pump and filter. (Photo: IIRS)

FOOD INDUSTRY

By way of example, special mention will be made of the role of analytical chemistry in the food industry. This is appropriate for two reasons. Firstly, it is in the area of food quality that the first legislation involving analytical chemistry was enacted and secondly, food production and processing is our major industry.

Prior to 1860 the adulteration of food was widespread. Sometimes it involved the addition of non nutritional materials (e.g. water to milk) but in other cases toxic substances were used. For example compounds of lead, arsenic and mercury were used to enhance customer appeal of confectionery! (11, 12). In response to this situation, anti-adulteration legislation was introduced in 1860 and 1875 (the 1875 act is still in force) and other legislation followed over the years. To-day, a whole series of regulations are in force controlling the use of additives and colours, permissible levels of contaminants such as toxic metals, pesticides, hormones, mycotoxins, etc. (see table 7) (11).

A significant development in the field of analytical chemistry was the establishment of the office of the 'Public Analyst' in 1860 (11, 12). Dublin incidently was the third city in the British Empire (after London and Birmingham) to appoint a Public Analyst (in 1862). This was in recognition that legislation controlling the composition of food was meaningless without the necessary mechanism of monitoring it. Each year, following the appointment of the Public Analyst, there were dozens of convictions in the Dublin Courts under the 1860 and later the 1875 acts. An interesting feature of law at that time was the power of a magistrate to order the convicted shopkeeper to publicise his conviction at his own expense by way of a newspaper advertisement. For example, the following advertisement appeared on the front page of the Irish Times of 27th February, 1972:

Adulteration of milk

At the Northern Police Court on Saturday, 17th February 1872, John Doyle, Dairyman, of 127 Dorset Street was fined five pounds for selling milk adulterated with 50% water.

Poor Mr Doyle had to pay not merely the fine but also the cost of the advertisement. Perhaps there is a lesson for our present day legislators!

To-day foodstuffs are analysed for purposes of

Checking compliance with legal requirements,
Checking compliance with customer specification,
Deciding the value of purchased raw materials, e.g. milk, sugar beet, grain, etc.

Most food processing plants will have the facilities for routine analysis of the foods which they process and a number of commercial laboratories offer services for this type of analysis. Some examples of what might be described as 'routine' analyses are given in Table 6. The more complex analysis of foodstuffs for contaminants, particularly at trace level (PPM to PPB), may be carried out by the larger processors but is more likely to be done by the Public Analysts, the State Laboratory, An Foras Taluntais, the Department of Agriculture and the IIRS. A list of some of the contaminants of significance is given in Table 7. For those interested in a review of modern methods of food analysis, reference 13 is recommended.

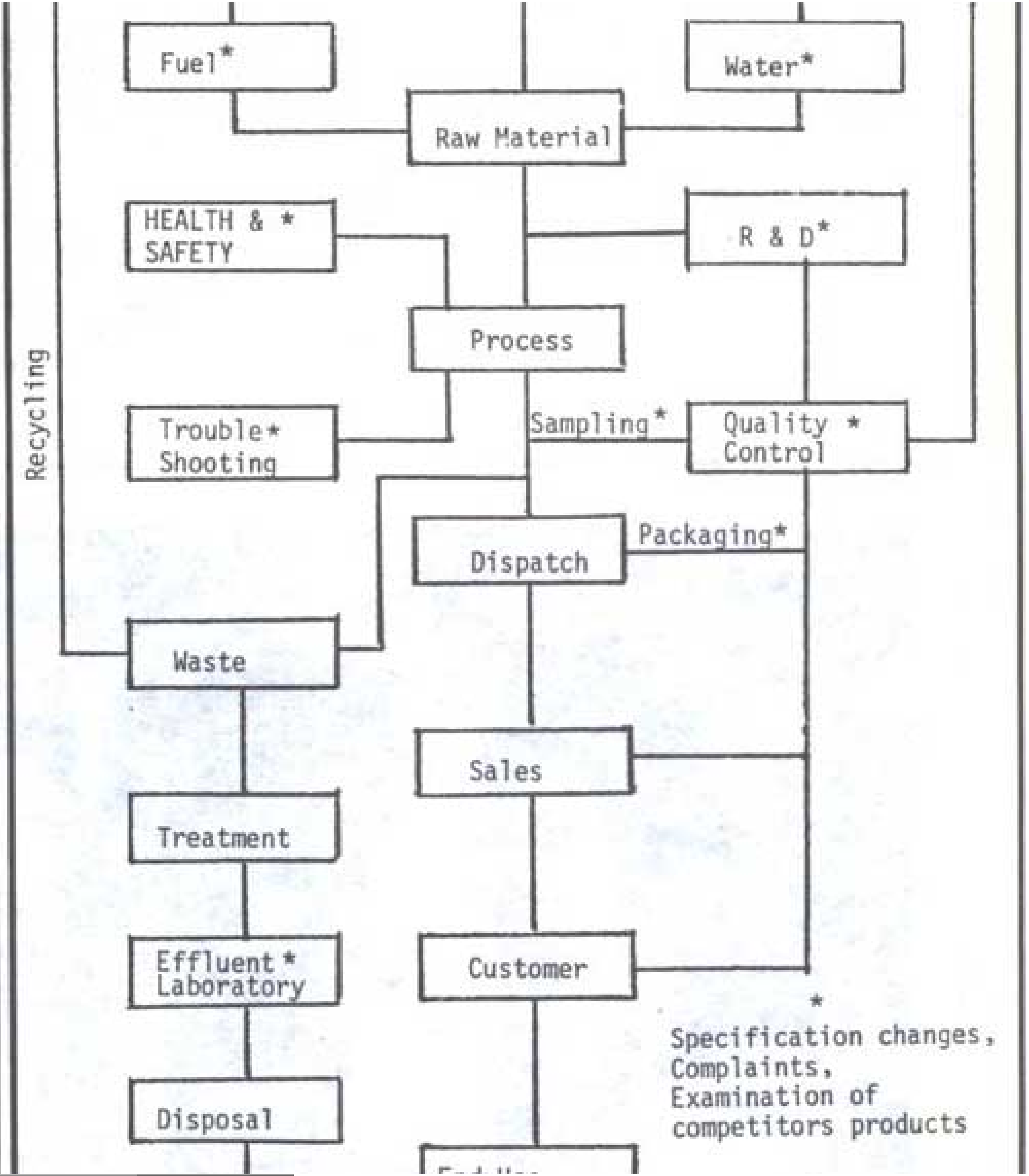
TABLE 6 SOME EXAMPLES OF ROUTINE FOOD ANALYSES

Milk	Fat, non fatty solids, antibiotics
Meat Products	Moisture, ash, protein, collagen
Cooking Oils	Saponification, free fatty acids, fatty acid distribution
Alcoholic Beverages	Proof content, solids, sugars, etc., SO_2
Composite Foods	Moisture, ash, protein, fibre, carbohydrate, minerals, vitamins, preservatives

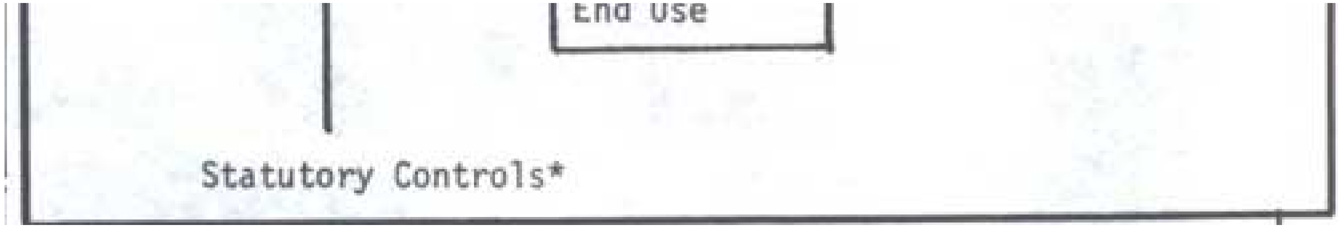
TABLE 7 SOME CONTAMINANTS IN FOOD

Arsenic
Asbestos
Artificial colours
Heavy metals (Pb, Cd, Hg)
Hormones
Mycotoxins
n-Nitroso compounds
Pesticides
Polycyclic aromatic hydrocarbons
Vinyl chloride monomer

FIGURE 2 INDUSTRIAL PROCESS SHOWING INVOLVEMENT OF ANALYTICAL CHEMISTS*



Fuel*
Water*
Raw Material
HEALTH & * SAFETY
R & D*
Process
Recycling
Trouble* Shooting
Sampling*
Quality * Control
Packaging*
Dispatch
Waste
Sales
Treatment
Effluent * Laboratory
Customer
*
Specification changes,
Complaints,
Examination of
competitors products
Disposal



INDUSTRIAL PROCESS SHOWING INVOLVEMENT OF ANALYTICAL CHEMISTS

THE ROLE OF ANALYTICAL CHEMISTRY IN THE ENVIRONMENT

Over the past ten years here has been a dramatic increase in public awareness in this country of the need for environmental protection. This has led to a number of direct and indirect actions by the authorities to ensure that at least all new industry and as far as possible old industry would comply with acceptable standards with regard to their discharge of gaseous, liquid and solid waste and their handling of dangerous substances. Some controls are achieved through direct legislation, others through conditions attached (a) by local authorities to the granting of planning applications and (b) to the awarding of IDA grants.

In all areas of environmental protection, the analytical chemist is intimately involved because almost every control or regulation relates to permissible concentration levels of substances which may be present in discharges, effluents, etc. Their analysis is therefore a prerequisite to the effective implementation of those controls and regulations.

Broadly speaking, the environment can be divided into two areas – the environment of the workplace (usually, though not always, indoors) and the general external urban or rural environment. Each has its own requirements and these will be discussed in the following sections.

THE WORK PLACE

There are three main areas of risk to workers from the use of chemicals. These are (a) direct contact with toxic, carcinogenic or corrosive chemicals, (b) risk of explosion, and (c) inhalation of airborne chemicals and dusts. Situation (a) requires negligible input from the analytical chemist; where the hazards associated with a chemical are known, a responsible management will provide protective clothing and training for operators and emergency facilities for dealing with accidents.

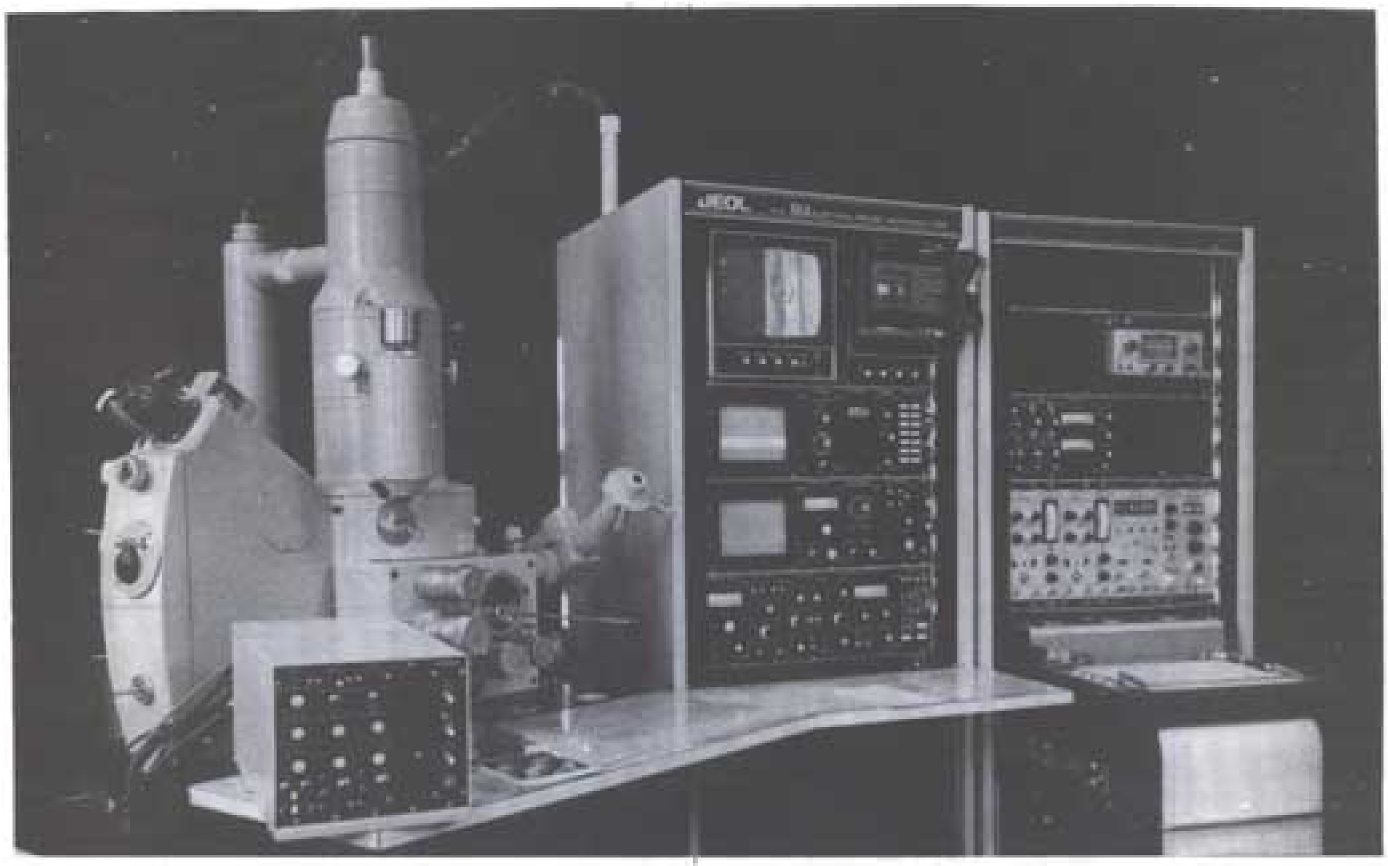
The prevention of explosions requires the monitoring of factory atmospheres for explosive vapours but this is usually done by non-analytical chemists using portable easy to operate ‘explosimeters’. These mainly operate on variations of the one principle whereby the atmosphere is drawn across a catalytically coated filament (or other device such as a pellister) and ignited. As a result, the filament temperature increases causing a change in its electrical resistance which is measured by making it part of a Wheatstone Bridge arrangement. The devices are calibrated to record (or alarm) when the atmosphere contains a flammable vapour approaching or in excess of its lower explosive limit (the lowest concentration which will sustain combustion). Examples of flash points and lower and upper explosive limits are given in Table 8 for some common substances.

TABLE 8 Flash points, lower and upper explosive limits (L.E.L. and U.E.L.) for some common substances

Substance	Flash point/°C	L.E.L./%v/v	U.E.L./%v/v
Methane	-------	5·3	14
Butane	-------	1·9	8·5
Petrol	-43	1·4	7·6
Acetylene	-------	2·5	81
Carbon disulphide	-30	1·3	44
Toluene	4	1·2	7·1

In some industries there is a legal requirement for a ‘Naked Light Certificate’ to be issued before ‘hot work’ (e.g. welding) can take place in certain areas. An example of this would be the carrying out of repairs to a tanker used for carrying petroleum products.

The third situation which requires the monitoring of airborne vapours and dusts involves the analytical chemist to a greater degree. Most common chemicals used in industry are rated according to what is known as their “Threshold Limit Value” or “TLV” (14) which may be loosely described as the maximum concentration of a dust, gas or vapour to which a worker should be exposed over a normal working shift. Exposures to levels at or below the TLV, on the basis of present knowledge, are not known to cause ill effect except in the case of allergy or hypersensitivity. The TLVs most commonly used are those published by an American group of government industrial hygienists (14) and these form the basis of the controls exercised by the department of Labour Factory Inspectorate in implementing the 1955 Factories Act and its many regulations. A list of TLVs for a number of substances are given in Table 9. The methods used to monitor factory atmospheres vary from simple hand operated pumps which draw air through an indicator tube (rather like the breathanalyser) to portable infra-red analysers. Gas chromatographic analysis of gases and vapours isolated in a variety of ways (such as absorption onto charcoal) is common in 'one-off' situations. Dusts are usually absorbed on membrane filters. Small battery operated 'personal monitors' are frequently worn by certain workers to measure actual inhaled levels rather than average concentrations for the whole work area.



Scanning electron microscope/electron microprobe analyser used for examination and chemical analysis of minute areas of solid surfaces down to 2-3 microns in diameter. This photo illustrates the complexity and sophistication of modern analytical equipment. (Photo: IIRS)

Pressure from trade unions, legislation and a sense of responsibility on the part of some employers has led to an increased amount of surveillance of factory atmospheres.

TABLE 9 Examples of threshold limit values (TLV's)

Substance	ppm (by volume)	mg/m^3
Ethanoic acid (acetic acid)	10	25
Propanone (acetone)	1000	2400
Carbon monoxide	50	55
Hydrogen sulphide	10	15
Ammonia	25	18
Sulphur dioxide	5	13
LPG	1000	1800
Silica		0·1
Asbestos	2 fibres/mL	

ETERNAL ENVIRONMENT

All new factories which received IDA assistance are required to meet minimum environmental standards set down by local planning authorities and the IDA itself on the recommendations of the IIRS. This applies to both air and water borne effluents. Legislation has also been introduced to protect the atmosphere and our lakes and rivers from pollution from whatever source it may arise.

AIR QUALITY

The oldest legislations in Ireland relating to the emission of harmful vapours is the Alkalie Act (1906), and this is still in force. Under it, a government appointed Alkali Inspector can set limits for the emission of acidic and other substances for scheduled industries. He is empowered to monitor the emissions and, if necessary, close down a plant which does not adhere to the limits he has set. It should be pointed out however that those powers are not frequently used. The only other national legislation relating to air quality are regulations, dealing mainly with the emission of black smoke, which were introduced in 1970 (15). A 1980 EEC directive on health protection standards for SO2 and particulate matter will, in due course, be absorbed into Irish law.

Other factors indirectly affecting air quality are the control of potential pollutants, e.g. lead in petrol and sulphur in gas/diesel oil and the occasional private legal actions taken against companies who cause pollution of the atmosphere.

Analytical techniques used to monitor air quality vary greatly and range from simple absorbers to sophisticated gas chromatographic and spectroscopic techniques (18). Sulphur dioxide can be simply measured by bubbling it through a solution of hydrogen peroxide (which converts it to H_2SO_4) and measuring the acidity produced. On the other hand, the measurement of air-borne asbestos at ambient levels (about 10^{-9} gr/m3) requires a sophisticated and tedious procedure using a Transmission Electron Microscope (19). Automated procedures for measuring many pollutants have been developed for continuous operation at unattended monitoring stations. Analytical research is continually going on to improve the detection limits reliability and ease of operation of the procedures used to monitor air quality.

WATER

Water, which is usually taken for granted, has a major part to play in determining the quality of our lives. Apart from that which we drink, it has a major amenity role and is used as a raw material in virtually every industrial process.

The discharge of unwanted waste by way of aqueous solution or suspension into sewers, rivers and lakes traditionally provided industry with the most convenient means for its disposal. The damage done to rivers and lakes in countries where industries developed in a haphazard and uncontrolled way during the industrial revolution bears ample testimony to this. Ireland is fortunate in that pollution of rivers and lakes is relatively rare and legislation introduced in 1977 is aimed at maintaining this situation (16). A number of EEC directives also are aimed at ensuring minimum standards for surface waters used for drinking purposes and bathing waters and these will in time become part of our national legislation.

A number of agencies are involved in various aspects of monitoring water quality including the Public Analysts, Local Authorities, An Foras Forbatha, IIRS, the Department of Fisheries, private analysts and industry’s own laboratories.

From an analytical point of view, routine water analysis is relatively uncomplicated and except where sample numbers necessitate automation, simple equipment and a minimum of expertise is required (See Table 10) (20). More complex procedures such as gas chromatography are required however to deal with pollutants such as pesticide residues or polycyclic aromatic compounds. Larger water laboratories use autoanalysers to deal with the throughput of large numbers of samples.

TABLE 10 Parameters monitored in water

Routine

pH	BOD	Total organics
Colour	Heavy metals	Turbidity
NO_2^-	Hardness	Fluoride
NO_3^-	Cl^-	Na^+
SO_4^{2-}	Cl_2	K^+
PO_4^{3-}	NH_3	Dissolved oxygen
Total solids	Conductivity	

Non-routine

Pesticide residues
Polycyclic aromatic compounds
Polychlorinated biphenyls (PCB's)
Unusual organic pollutants e.g. acrylonitrile
Asbestos fibre

Sampling is an important part of water analysis, particularly in carrying out surveys of rivers, lakes and tidal estuaries. Due account must be taken of river flows, tidal currents and still water areas in assessing where to sample and how to interpret results.

CONCLUSION

In this article, an attempt has been made to highlight the multi aspect role of analytical chemistry in industry and the environment. The need for analytical chemistry stems from the necessity to quantify chemical composition for purposes of process and quality control and the implementation of legislation. It is not surprising therefore that more graduate chemists are involved with analytical chemistry in their working careers than all other aspects of the subject. This was shown in a survey carried out in 1974 (17) and while the data is admittedly out of date there is no indication that the trend has altered in the meantime.

It can be seen that analytical chemistry, unlike other branches of the subject, is very much involved with the everyday functioning of our society, whether it be in facilitating trade or in the implementation of legislation. More often than not, the analytical chemist in these situations will be dealing with non-scientific persons such as lawyers, judges, legislators, civil servants, purchasing managers, etc. Apart therefore from technical competence, he must be able to communicate with those outside his own profession. Often this is not easy. Concepts such as ‘detection limit’ or the statistical tolerances associated with results obtained by a particular procedure can present amazing difficulties, particularly when the outcome of a dispute or the value of a shipment of goods may depend on them.

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“Scientific literature rides the caboose on the train of science”
Nature editorial 24/4/81

“*I think it was in a course in quantitative chemical analysis that an appreciation of the scientific method and its rigours began really to take hold of me…*
There were no short cuts to beat clear thinking, careful technique and endless patience. Later on, I found that the same unnatural methods are always required in those activities commonly called ‘research’”.
Robert S.Mulliken
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