

SURVEY OF CHEMICAL RESEARCH AND DEVELOPMENT IN CANADA

" ANALYTICAL CHEMISTRY "

COMMITTEE NO. 1 REPORT

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ANALYZED



## REPORT ON RESEARCH AND DEVELOPMENT IN ANALYTICAL CHEMISTRY IN CANADA

### Definition of Field

It is difficult to agree on a precise definition of the field of analytical chemistry. In essence, it is concerned first with what substances - elements, compounds, isomers, etc. - are in a given specimen of material, and second, proportions in which one or more of these are present. The activities of analytical chemistry can therefore be grouped under certain general headings of which the following is an attempted summary.

- a) Identification - By comparing the properties, physical or chemical, of a given material or portion thereof with those of known substances, identification or detection of the presence of a given substance may be established. Traditionally this part of chemical analysis has been described by the adjective, qualitative.
- b) Measurement - Although qualitative analysis may involve measurements, for instance, melting points or refractive indices, measurement in analysis more often applies to determining amounts of a given substance in a sample. This part of an analysis is described by the adjective, quantitative. The quantity measured may be a property of the sample itself or a portion of it, or it may be a measurement applied to a reaction in which the material under examination takes part.
- c) Separation - For both qualitative and quantitative analysis to be effective, it is necessary to isolate one or a small group of

constituents in a material and chemical analysis has traditionally been concerned with effective separations as complete as practicable among the substances present in a given material.

- d) Evaluation - An important consideration in analytical chemistry is, or ought to be, the evaluation of the information obtained by one or by a variety of methods. This may include a statistical examination of results obtained on samples by a particular method of analysis, or it may involve comparing a variety of methods for carrying out analysis for a particular constituent. It presupposes the existence of suitable standards against which analytical methods may be compared or evaluated.

The foregoing describe operations that are typical of chemical analysis but in order that analytical operations may be carried out meaningfully, research in the field of analytical chemistry has been conducted along lines that are not necessarily obviously related to the practice of analysis itself. Thus to the foregoing we add . . .

- e) The Study of Analytically Useful Reactions - For example, the extent, the rate and the mechanism of the reaction, the nature of products and side-reactions, the factors that govern and affect the sensitivity and the selectivity of analytical reactions and methods, and so forth. This type of study is invaluable to the development of new procedures and improvements in existing procedures.
- f) Method Development - The development of new procedures for a particular analysis, or the modification of existing procedures to suit a particular need.

Considerations Affecting the Definition of Analytical Chemistry -

One aspect of the subject has a decidedly applied nature, in that its methods and techniques are widely applied not only to the other areas of chemistry but to several areas of science, for example, chemical and metallurgical engineering, geology and geochemistry, biology and biochemistry, medicine, physics, geography, and so forth. The practice of analytical chemistry is spread over a vast area of experimental science. Its techniques and methods are utilized as means to specific ends, the ends in these cases being the procurement of data by analytical methods to advance knowledge in another area of science. This ubiquitous nature of analytical chemistry has led to the impression that its role in science (and particularly chemistry) is strictly a supporting one - one that provides a service. This impression is strengthened by the fact that industry relies so heavily on the methods of analytical chemistry to provide control checks on the quality of products and data for the solution of non-analytical problems.

A second aspect is concerned with the more fundamental or basic nature of analytical chemistry at the level of research. Because of the vital nature of analytical chemistry as an applied science, this aspect has been overshadowed by the first and, indeed, has often been entirely overlooked. The fact is, however, that a valid and significant area of chemical research can be associated with analytical chemistry, and that innumerable areas in the field of chemical analysis offer opportunities and requirements for basic research. As mentioned above, research in analytical chemistry may involve obtaining knowledge concerning the thermodynamics, kinetics, and mechanisms of analytical

reactions whether these are characteristic of the classical solution processes of titrimetry and gravimetry, or of processes associated with "modern" methods of analysis. The latter include atomic absorption and flame photometry, spectrophotometric processes such as charge-transfer or fluorescence in the ultra-violet or infra-red regions, or electrochemical processes, or chromatographic processes, and so forth. Knowledge of this kind not only advances our understanding of the fundamental nature of chemical processes but aids immeasurably in the development of new more sensitive and more selective analytical procedures, or in the improvement of existing procedures.

A number of respondents, who have provided the committee with much of the information upon which this report is based, have stated that it may often be difficult to identify research activity in analytical chemistry because the latter is only seen as an applied branch of the subject. Many scientists in this country have in the past assumed a pose of down-grading research in an applied part of science as contrasted with research in a pure area. Because of the stigma attached to research activity in an applied area, some people may identify their research with a field such as inorganic chemistry or physical chemistry when perhaps it should properly be related to analytical work.

In addition to the foregoing difficulty, the committee believes that a great deal of analytical work is carried out under the umbrella of research activity in neighboring fields of chemistry. Thus it is unlikely that much quantitative work can be carried out in an area such as physical chemistry without appropriate methods of analysis being available. In many instances, because of the original

nature of work being conducted, the researcher may have to adapt existing chemical methods or even invent new chemical methods of analysis. In a survey of the scope of analytical research and development, it is probable that many projects will be overlooked which contain a significant component of analytical activity because they are identified with the primary objective of the research, which may be in chemical engineering or in many branches of pure chemistry.

These comments are bound to create the impression that a significant amount of research, and especially development work, may not be credited to analytical chemistry that perhaps should be. However, in connection with this survey another factor will tend to exaggerate the scale of analytical chemistry research and development. The areas of interest with which projects or groups are to be identified are shown on pages ~~to~~ of this Report. Analytical chemistry under this classification embraces the general areas numbered 011 and 012. Within these area classifications are listed a number of techniques; for instance under 012 are listed spectroscopic techniques. The authors know for a fact that a number of spectroscopy projects, the intent of which could in no way be regarded as analytical, have been reported in the C.I.C. Survey under this category. This illustrates perhaps the greater appropriateness of classifying research by objectives rather than by techniques. Some further evidence on this point appears under "Scale of Activity" later in this Report.

Changes in the Nature of Analytical Chemistry - A number of technological developments have had a bearing on the nature of chemical analysis as practised today. For example, within about one generation of

chemists there has become available a vast array of sophisticated instrumentation virtually undreamed of 25 years ago. Almost all major laboratories engaged in research or development work in chemical analysis must be provided with a variety of instruments and devices for carrying out physical measurements and for recording or otherwise processing the numerical output for these instruments. The following list of methods, while not exhaustive, will furnish some idea of the range and complexity of contemporary analytical skills.

- (a) Spectroscopy
  - (i) Emission - Photographic recording  
- Direct reading
  - (ii) Atomic Absorption
  - (iii) Flame Photometry
  - (iv) Ultraviolet and Visible -  
Single reading or recording
  - (v) Infrared - recording
  - (vi) Raman
  - (vii) X-ray fluorescence
- (b) Mass spectroscopy
- (c) Magnetic Resonance Spectroscopy
  - (i) Nuclear Magnetic Resonance
  - (ii) Electron Spin Resonance
- (d) Thermal Transitions
  - (i) Differential Thermal Analysis
  - (ii) Thermogravimetric Analysis
- (e) Chromatography
  - (i) Adsorption methods
  - (ii) Ion-exchange methods
  - (iii) Partition methods (especially gas-  
liquid Chromatography)
- (f) Activation Analysis

- (g) Electrochemistry
  - (i) Controlled electrodeposition
  - (ii) Coulometry
  - (iii) Polarography

Specifically among such devices mention might be made of the present availability of commercial Raman spectrophotometers utilizing photomultiplier detectors and, in increasing numbers, laser beam sources of illumination. The application of Raman spectrophotometers for chemical analysis is becoming increasingly common.

Enormous improvements in devices measuring radioactivity, and the relatively easy access to sources of high-intensity neutron flux have made possible the exploitation of neutron-activation analyses as a powerful tool for quantitative and qualitative analysis at very low concentrations. Tremendous strides have been made in the separation of organic compounds by the use of gas chromatography and vapor-phase chromatography, and such techniques have opened up possibilities of analyses that might at one time have been considered impractical. The widespread use of magnetic resonance devices (NMR and ESR) has introduced possibilities for conformational analysis in organic compounds and a host of other applications of interest to the analyst.

The availability of machine computers has provided the possibility of more sophisticated numerical analyses of analytical results leading to an expanded application of statistics to chemical analysis. It is now possible to adapt many analytical instruments to provide digital outputs enabling the instrument readings to be immediately available for mathematical analysis by machine computer. It is probable that research in continuous systems of analyses may be spurred on by the availability of analog computers which can simulate process devices.

A number of practical considerations have given rise to needs for analysis to be carried out for very small amounts of the constituents sought expressed as either absolute amounts or concentrations. To give a specific illustration, the preparation of materials as solid state devices requires careful knowledge and control of the presence of traces of foreign elements; analytical methods suitable for such materials, and for the determination of minute traces of elements in them, have imposed new demands on the chemical analyst.

The nature of the instrumentation required for these physical methods creates certain problems, first, in the high costs of procurement and maintenance, and second, in the skills and versatility of the personnel using them. Under any system of cost accounting analysis is becoming expensive, and it is a recurring responsibility for laboratory directors to maximize the return on the investment in equipment and manpower. Clearly this will only be achieved if analytical personnel have the skill and discretion to utilize their tools to best advantage. One area of concern to industry here is the additional cost applied to items of expensive equipment by the imposition of taxes and duties in this country. Nearly all such equipment has to be imported since no significant domestic production exists, and the validity of tariff duties applied to the basic cost has been questioned.

There is some evidence that research and development in chemical analysis has been uneven in Canada in the sense that full advantage of new technology has not always been taken, but more

important because personnel who have an adequate and broadly based knowledge of the possibilities of the newly available instrumentation are not available to mount programs of research. In this connection a number of comments about the training of persons in analytical chemistry will be given in a later section of this report.

To indicate more clearly the range and distribution of techniques practised by analytical chemists, we draw upon the results of a survey conducted in the U.S.A. by the National Science Foundation. No comparable data for Canada are known to the authors. The American study identified 20,500 analytical chemists in 1966, representing 16.4% of all chemists surveyed. The distribution of their areas of specialization are given in Table 1 (reproduced by permission of the editor of Chemical and Engineering News). (page 10).

The relevance of these data to a survey of research and development in Canada is possibly open to question, first, because many analytical chemists are engaged in non-research activities such as process control or product testing, and second, because the relatively higher investment in research in the U.S.A. as compared to Canada may skew the distribution toward more sophisticated techniques in that country. However, the information in this table discloses something of the relative importance to analysis of the techniques listed.

A Summary of Current Research and Development Activity in Canada -

(a) Universities - In those universities where research in analytical chemistry is going on, by far the most evident emphasis is on studying the fundamental chemical reactions utilized in wet chemical separations and determinations. One laboratory is

TABLE I Specialty areas of analytical chemists

Specialty	Per Cent	Estimated Number Of Chemists
Absorption spectroscopy	17.5	3,600
Biochemical analysis	4.4	900
Chemical microscopy	0.7	100
Chromatographic analysis	15.9	3,300
Distillation analysis	0.4	100
Electrochemical analysis	5.0	1,000
Electron Microscopy	0.7	100
Emission spectroscopy	5.7	1,200
Extraction analysis	1.2	200
Fluorometry, phosphorimetry, and Raman spectroscopy	0.6	100
Gas analysis	2.2	500
Gravimetry	8.3	1,700
Magnetic resonance spectroscopy	2.6	500
Mass spectroscopy	2.9	600
Microchemical analysis	3.7	800
Nucleonics and radiochemistry	4.7	1,000
Qualitative analysis	5.8	1,200
Titrimetry	6.8	1,400
X-ray and electron diffraction	2.8	600
Other	7.9	<u>1,600</u>
TOTAL		20,500

making significant contributions to developments in gas chromatography. Another is active in studying fundamental characteristics of absorption spectroscopy.

There appears to be little or no teaching or research in instrumentation (as distinct from instrumental analysis) and where it exists it is more apt to be in departments of chemical engineering related to control systems. One university has introduced (but not yet taught) courses in analytical instrumentation to undergraduates.

- (b) Industry - Detailed information is not broadly available, but based on a limited number of submissions and personal knowledge of committee members, the following comments appear appropriate. There is very little research work in this field, but a substantial amount of development work, especially in the adaptation of methods to specific problems. Although analyses proper are commonly performed by technologists, a supply of scientists is required whose prior training enables them to take full advantage of the new developments in instrumentation. Since many industrial analyses are of the continuous monitoring type, a close relationship must be developed between the analytical chemist and those responsible for process control.
- (c) Government - There is evidence of a healthy state of analytical development work in many government divisions. For instance one laboratory states that half of its staff is engaged in development work in analysis; and another suggests that one-third of its staff time is devoted to analytical work even though some

of this may be devoted to research projects bearing other labels. Most of the work in government laboratories is problem-oriented, though a higher proportion of pure research work is evidently done in these than in industrial laboratories. Government departments are conducting a significant amount of publishable research in analytical chemistry in such diverse fields as forest products, hygiene and clinical chemistry, food and drugs, agriculture, bio-sciences, and numerous others. In the submissions from all of these, stress is placed upon new developments in instrumentation and automation, and for expanded training of university graduates to enable them to meet the challenges of analytical problems. The points are made, for instance, that scientists, other than chemists (e.g., biologists), are frequently called upon to do analytical work and rarely have the background to do it. Also that many graduate chemists have had inadequate exposure to practical laboratory work to have developed the necessary skill and judgement to tackle analytical problems by a range of techniques.

#### Scale of Activity

The scale of research and development activity in analytical chemistry has been surveyed through the C.I.C. questionnaires within the areas of chemistry designated by the code numbers 011 and 012 (see page 7 for classification). If all the activity classified under these headings is truly analytical chemistry, one would be clearly entitled to assume that this field is active and well supported. For instance, of the operating

expenditures for research and development detailed in Table 2, the amounts for CIC areas 011 and 012 expressed as percentages of the total operating expenditures for all C.I.C. code areas were as follows:

Universities	7.3%
Industry	6.1%
Government	10.1%
Over-all	7.0%

There are grounds for doubting, however, whether the scale of expenditure suggested by Table 2 is actually applied to work which is normally regarded as analytical chemistry. For instance, C.I.C. code area 011 includes nucleonics and radiochemistry, and one might reasonably wonder whether the support indicated for this (including \$1.263 million by the federal government) may not include work properly assignable to Nuclear and Radiation Chemistry, Committee 12. Similarly, there is evidence that considerable work reported under spectroscopy (area 012) was regarded by respondents to questionnaires as properly belonging under a different category (such as Physical Chemistry). These examples illustrate the difficulties inherent in eliciting information by questionnaire.

Particularly suspect, indeed, are the figures relating to university support. These show operating expenditures in Table 2 amounting to nearly \$1.5 million for research and development. In addition, returns to the questionnaire circulated to university departments show over \$1 million investment in major equipment, plus a major installation item of \$100,000. This level of expenditure is simply not consistent with the amount of activity

TABLE 2

Operating Expenditures (1966/67) for Research and Development

CIC Codes 011 and 012

	<u>\$ 000</u>			
	<u>Basic</u>	<u>Applied</u>	<u>Development</u>	<u>Totals</u>
011 Universities	524.3	79.7	10.9	615.0
012 Universities	823.1	37.7	11.2	872.0
011 Industries	352.0	<del>2160.6</del>	1239.7	<del>3752.3</del>
012 Industries	339.9	878.5	582.0	1801.2
011 Government	<del>342.1</del>	404.8	1351.0	<del>2097.9</del>
012 Government	<del>249.0</del>	92.0	34.0	<del>375.0</del>
				<u>9513.4</u>

TABLE 3

Effort in terms of Manpower in University Departments

CIC Codes 011 and 012

	<u>Academic Staff</u>	<u>Postdoctoral Fellows</u>	<u>Graduate Students</u>	<u>Technicians</u>	<u>Totals</u>
011	46	13	80	37	176
012	53	23	95	20	191
011 *	28.5	11.1	51.7	28.9	120.2
012 *	26.8	12.4	42.3	14.4	95.9

\* Figures adjusted to represent full-time equivalent effort in R. & D.

in analytical research work known by members of this committee to be going on in the universities. This can be illustrated by the following data. From the N.R.C. Annual Report on Support of University Research (1966-67) the committee were able to identify only 23 awards of operating grants (out of 494 in chemistry) to faculty members whose research was recognized as being in analytical chemistry. The value of these awards was very close to 3% of the total of the operating grants. Similarly there appeared to be 3 major equipment grants, amounting to just over 2% of the total value in this category, and to 4% of the number of such awards.

There are comparable discrepancies in the assessment of manpower engaged in analytical research and development in the universities. For instance Table 3 shows numbers and categories of manpower reported by university departments to be engaged in this area. These data are taken from returns to the questionnaire submitted by the C.I.C. survey. However, the total number of graduate students in analytical chemistry reported by N.R.C. is 71, (Graduate Students at Canadian Universities. Pub. 9340, 1967). The larger numbers of graduate students reported in Table 3 must be engaged in research identified by the categories 0200 to 0219 which is nevertheless not seen by the responding departments as analytical chemistry per se.

#### Character of Research Activity

The responses to the project questionnaires from government departments provide the only evidence concerning the level of activity among the various specialty classifications 0200 to 0219. One quarter of the projects reported were assigned to category 0219 -

that is, these could not be assigned to any of the nineteen named specialties, and this group represented 15% of the government operating expenditure in this area. By far the largest expenditure (51% of the total) was reported under category 0215 - Nucleonics and Radiochemistry - and yet this category accounted for only one-sixth of the number of projects reported.

More than one-fifth of all projects reported in this area were in biochemical analysis, although the expenditure incurred for these was only 3.2% of the total. Spectroscopy (0200 and 0209) accounted for another 20% of the projects reported with an operating support nearly 8% of the total. These, together with chromatography (0203), make up the great bulk of the specialties reported by the government laboratories, and account for two-thirds of the reported expenditures.

As the data of Table 2 reveal, by far the bulk of the university expenditure in this analytical area is for basic work. The work in the government laboratories appears to be strongly supportive of development work, but the data are seriously skewed by the very large expenditure (\$1 million) shown for development work in nucleonics and radiochemistry (0215). Apart from this particular specialty the government laboratories support basic research, applied research, and development, in diminishing degree. Industrial expenditure on basic research in the areas covered by this section is understandably rather less in relation to applied work and development.

One final item from the project questionnaires returned by government laboratories should be mentioned. Respondents were

invited to indicate which, if any, of the areas of specialization, 0200 to 0219, should receive more attention in Canada. In terms of the numbers of neglected specialties analytical chemistry ranked third (after biochemistry, and agricultural and food chemistry) among the 20 area committees. The most frequently mentioned specialties in this response were absorption spectroscopy, mass spectrometry, and x-ray and electron diffraction.

Short Range Forecast of Development in this Field -

There is evidence of a shifting climate of opinion toward research in applied science in Canada. On the basis of this, the committee feels that a prediction is warranted that there will be an increase in research and development in analytical chemistry, and that this increase will be most visible in the universities and government research laboratories. The possibility of a number of research institutions being set up on university campuses appears to be a logical evolution of the expansion of graduate work in science in the Canadian universities. It is probable that such institutes will be devoted in most instances to applied activities and that analytical chemistry will form a significant component of the activity in such institutes. Such a prediction can be justified, at least in part, because of the interdisciplinary nature of applied chemical analysis.

It also seems probable that given the foregoing developments, universities will increase the amount of analytical chemistry in undergraduate programs and correspondingly increase the activity of the graduate level. It is expected that a number of Ph.D. - trained analytical chemists will be required to staff university departments and departments in community colleges within the next few years.

It is widely held that a shortage of personnel possessing the knowledge to exploit modern instrumentation for analytical purposes exists in this country to the extent that this is an impediment to the expansion of research and development in this area. The problem must be attacked at the source by encouraging university departments to develop more activity in analytical chemistry. It is evident that for a student to become familiar with the applications of modern instrumentation for analysis he must have opportunities to use such instrumentation in a meaningful way. This presupposes that such equipment will be available in the universities and that students will have access to it. There is some belief that this is not at present always the case. There exists another and virtually untapped method of exposing university students to a wide range of analytical problems and the equipment currently available to solve these through much increased cooperation between universities and industrial laboratories. A realistic approach to the problem of an acute shortage of adequately trained personnel would suggest that an increased exposure for university students to practical chemistry in industrial laboratories would be highly desirable and that university faculty should take the initiative to try to establish such cooperation.

One of the most important requirements for any laboratory involved in development of new analytical methods, or evaluation of several methods, is the provision of standard samples. The preparation of these is time-consuming and often uneconomical for a single application. There is a strong case for the establishment of a Canadian Bureau of Standards for Chemistry and Physics; this could be

a government agency like the National Bureau of Standards in Washington, or a private activity like the Bureau of Analysed Samples in the U.K., although the sustenance of a private organization might be marginal within the present Canadian economy.

Handicaps to Research and Development -

a) Equipment: There is no clear-cut evidence from our enquiry that a need is felt for greatly increased provision of analytical instrumentation. One of our respondents has pointed out that much of the expensive instrumentation now available for analytical research and development could be used more efficiently and could provide much improved information by more careful mathematical analysis of the output data. There is no doubt but that a demand for more and more sophisticated equipment will arise to meet specific analytical problems. The availability of such expensive equipment will be controlled by its cost and a situation may ultimately develop in which particular kinds of analyses can only be performed in certain laboratories in the country. However as long as a suspicion lurks that existing equipment is not being exploited to the fullest possible extent, it can scarcely be claimed that this is a major impediment to the expansion of research and development. As mentioned previously in this report, there is an expanding need to relate the output of analytical instrumentation and the assessment of analytical results to electronic computers. We have had no indication, however, that the availability of computational facilities in universities and major industries is insufficient to meet these requirements for analytical chemistry.

b) Library Facilities - No particular concern was expressed by our respondents concerning the adequacy of library facilities in this field. The view was expressed that a Canadian journal for analytical chemistry or a section in the Canadian Journal of Chemistry dealing with analysis might serve as a single vehicle for publication of a variety of research and development reports in this field based on Canadian work. However the authors of this report are inclined to believe that this suggestion may be premature.

In common with most areas of scientific investigation, analytical chemistry is plagued by problems relating to information retrieval but there is no indication that the situation is more serious in this field than in any other.

c) Manpower - Throughout all the responses to our enquiry, there is consistent concern over the lack of university trained personnel capable of carrying out research and development in analytical chemistry. This complaint does not extend to the supply of technologists, of which there appears to be an adequate supply for routine work or development work under supervision.

The conclusion is inescapable that the universities have neglected analytical chemistry as a discipline requiring theoretical and practical training, and that in consequence, university graduates are unable to take full advantage of the potential resources for carrying out analytical work. In the majority of universities analytical chemistry is offered once in the undergraduate syllabus. The course in many cases is not

taught by an analytical specialist, and usually occurs too early in the degree program to be able to make full use of the principles of physical chemistry or of physics on which many modern analytical practices are based. The content of such undergraduate courses can be judged by a sampling of the text books from which they are taught; and from these it would appear the teaching is mainly in classical wet inorganic chemistry analysis, with only a token exposure to simple instrumental procedures such as potentiometry and colorimetry. Given this account of formal training in analytical chemistry, the student is expected to pick up any other knowledge about analysis through courses in inorganic, organic or physical chemistry, or even in physics.

This situation has led to a shortage of personnel competent to direct or organize activity in analytical chemistry. It has tended to result in a certain number of people trained in other fields such as Biology, Geology, Physics, etc., being obliged to assume the role of analytical chemists. Because problem-solving in analysis frequently requires familiarity with a variety of experimental approaches, it is considered that a healthier approach to the manpower problem is to have personnel trained in the universities in an assortment of analytical techniques with some competence and judgement to evaluate their application to particular situations.

RECOMMENDATIONS

The committee recommends in summary:

1. That increased prominence be given by universities to analytical work in the undergraduate curriculum and that postgraduate training in analytical chemistry be increased. We feel that the analytical chemistry taught to undergraduates should be more in accord with contemporary practice.
2. Increased cooperation between universities and industry and government laboratories, through which students might be able to encounter analytical problems and analytical devices through visits to, or employment in, non-university laboratories, and through universities availing themselves of resource personnel from government or industrial laboratories to give lectures in specialized subjects.
3. That universities attempt to provide short courses for practising chemists in which modern developments in analytical practice can become more widely known.
4. That consideration be given, perhaps through the National Research Council, for the establishment of a Canadian Bureau of Standards.