CONTRIBUTIONS OF RADIOCHEMISTRY AND NUCLEAR ANALYTICAL TECHNIQUES TO SOCIETY AND TECHNOLOGY: SOME EXAMPLES OF 35 YEARS' EXPERIENCE IN DELFT

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1. INTRODUCTION

Budget cuts and the oscillating public debates on the benefits of nuclear research reactor facilities are a continuous threat to many nuclear institutions. University affiliated institutions often face additional problems if their facilities offer sufficient scientific challenge and if an education in nuclear sciences provide students a sound outlook for a professional career. Such discussions may be an excuse for non-fulfilment of vacancies and reduced priority in the academic curriculum. Universities tend to reallocate their funds to more contemporary sciences such as molecular biology and nanotechnology. Their choices are based on relevance, quality viability and productivity. Relevance is often measured on the basis of external funding of research programs as well as on career opportunities for students. However, the areas with opportunities for external funding are largely determined by socio-economic developments, sometimes even at the (inter)national political level. The thematic areas in the scientific Framework Programs of the European Union [1] illustrate this.

The relevance and viability of an expensive facility like a nuclear reactor centre can also be demonstrated by making the facilities available to provide measurements on request by outsiders. Ideally, such services should be sustainable, but even the existence may lead to consider the centre being of regional or national importance.

Nuclear reactor centres have various options for such services, like production of radionuclides; neutron transmutation doping; neutron activation analysis; neutron radiography and others.

This contribution gives examples of the typical analytical services provided by the Interuniversity Reactor Institute of the Delft University of Technology. This institute houses the only university research reactor in the Netherlands, a 2 MW swimming pool reactor, with associated facilities for neutron research, neutron activation and radiochemistry. Within the services provided by the Radiochemistry Department, emphasis is given to neutron activation analysis and the radiotracer method.

2. HISTORICAL BACKGROUND

The Radiochemistry Department has a long history and culture in making their facilities available to outsiders. As from the start of its operations, in the early 1960s, the policy of the institute was to demonstrate the opportunities of radioactivity and nuclear measurements to scientists in the applied fields. This approach showed to be more successful for radiochemistry than for reactor-physics and neutron-physics. Initially, academic collaborations were pursued, but rapidly also governmental organizations showed interest in the use of, e.g., radioactive tracers, measurement of natural radioactivity and neutron activation analysis. The use of radiotracers was advocated in the academic curriculum, resulting in much collaboration within the faculties of chemical engineering and applied physics with the university.

3. NEUTRON ACTIVATION ANALYSIS

Neutron activation analysis, which during the end of the 1960s and early 1970s was the only multi-trace-element technique, rapidly found interest at many other universities. The growing number of requests for analyses in Delft evoked the development of a 'laymen's system' for INAA **Error! Reference source not found.** in which the students from the other university faculties in applied fields such as geology, geochemistry, environmental sciences and biology, were trained to perform their own analysis using the institute's facilities without having a specific education in radiochemistry or nuclear physics. Initially this was done cost free, but the growing demand also implied an extension of the gamma-ray spectrometers. This marked the onset of paid analysis, even by the universities, although initially this was often realized by payment '*in natura'* via the procurement of detectors or spectrometer components.

Via the contacts with various governmental organizations also a network developed with links to the private sector. The 'Cd decree,' a national regulation in the mid-1980s following EU directives, caused such a growth in demand for determinations of Cd in plastics using INAA that a business-type unit was created with permanent additional personnel performing the analyses and managing the requests and accounting. The staff involved were trained in commercial communications and several advertising campaigns were initiated to improve the visibility. The income from these analyses was earmarked for the INAA group, providing the opportunity for improvement and further extension of the NAA facilities. One important spin-off of the contacts with customers was that many of the advantages of INAA seldom appear to excite customers. As such, new strategies for marketing had to be developed. Moreover, it became necessary to improve the efficiency, effectiveness and quality of these services. This culminated in the EN 45001 accreditation of the quality system by early 1993, later followed by ISO/IEC 17025 accreditation. The services require a high degree of automation such as sample changers and dedicated software, and parallel measurement systems. The automation stretches further than just sample changers. Already by the end of the 1970s the INAA software had been developed in such a way that a full gamma-ray spectrum analysis and interpretation towards element concentrations could be realized in about 1 minute per spectrum [2]. In addition, software was developed to schedule the measurements on the sample changers so as to measure 24 h/day, 7 d/week. Moreover, the software allows for different counting times of samples and flux monitors, and generates and interprets automatically quality control charts.

However, operating the software as a 'black box', which basically is possible, will inevitably lead to wrong results. Building technically competence of personnel with the ability to optimize analytical protocols and with insight in possible sources of spectral interference and other sources of error may require typically at least 5 years of full commitment to INAA and gamma-ray spectroscopy. The facilities for INAA and the quality system of the laboratory have been described elsewhere [3][7].

The organization of the INAA services has changed several times in the past decade as a result of changes in the Institute's policy. At the start of the 1990s, a commercial business unit was established, consisting of a marketing and public relations manager, administrative support and three technicians. All salaries were covered by the income from the analyses. This business unit worked in liaison with the nuclear analytical methods research group, which was responsible for the operational availability of the facilities and the analytical quality of the results. The laboratory for INAA identified external and internal customers. Scientists from other universities or research establishments, governmental bodies and industry form the first category. The internal customers were scientists within the mother institute, mainly from the Radiochemistry Department. Some of these internal customers were trained by the laboratory to carry out the analyses on their own. The external customers were fully charged for the analyses whereas the internal customers only paid for the consumables, such as capsules and internal quality control samples. The remaining revenue of the analyses remained earmarked so as to facilitate maintenance, replacement and extension of the facilities.

The advantage of this structure was that the research group was not hindered by the paradox of priorities for research or for satisfying commercial customers. A disadvantage was the absence of technical knowledge with the business unit manager, requiring regular back-up by the research group for advises on the feasibility of analysis. The university's public relations department assisted in the development of brochures and material for boots at exhibition. One important spin-off of the contacts with customers was that many of the advantages of INAA seldom appear to excite customers. As such, new strategies for marketing had to be developed.

By the end of the 1990s, the management of the Institute did not allow fulfilling vacancies in the administrative support and the management of the business unit, and the unit was merged with the nuclear analytical methods group. Moreover, the University decided that providing commercial services was not acceptable for an academic institution, with exception for those services that could be considered as unique for the reactor centre. A financial evaluation by the university accountants resulted in an estimate of an analysis cost that was a multifold of the cost, estimated by the Institute itself. The University considered that there was no viability for commercial services, and the institute's management decided that NAA services could only be provided primarily for scientific collaborations rather than to serve industry and governmental institutions. Consequently, a network of industrial customers was replaced by a network of academic institutions. However, academic institutions seldom are able to pay the full cost price of services but the deficit was allowed to be accounted for by co-authorship in resulting scientific publications.

The advantage of this structure was that a network of scientific collaborations could be developed, resulting in several jointly acquired research grants and various scientific publications, by which also additional budget was obtained. It should also be noted that in research grants lead to a higher academic respect that third party income. A disadvantage was the increased paperwork since also all accounting and client relations had to be maintained by the research group.

By 2005, the policy of the Institute had changed again because of budgetary problems. The value of scientific publications is no longer considered any more in the turnover of the NAA services. A new cost analysis has been carried out based on a different view to the contribution of the depreciation of equipment, resulting in a cost price much lower than prescribed five years earlier by the University. Also a new structure has been designed, in which the NAA services will be completely separated from the nuclear analytical methods research group. Marketing and accounting will again be done by a separate manager with background in physics rather than in chemical analysis. The focus will be again on the industrial market rather than on collaboration with academic organizations.

Characteristics of the various organization structures have been summarized in Table 1. It should be noted that short-term policies as described above, often characteristic for university environments, are detrimental for the motivation of personnel, for maintenance of a network of customers (both industrial as academic) as well as for the credibility of the marketing.

TABLE 1. SUMMARY OF ADVANTAGES AND DISADVANTAGES OF VARIOUS ORGANIZATION STRUCTURES FOR COMMERCIAL APPLICATIONS OF INAA AT IRI, DELFT

Organization structure	Advantages	Disadvantages
1975-1990 Laymen's system	Easy access, no paperwork, no additional personnel needed, no marketing needed, payment *in natura'*	Training, control of analytical quality, sustainability, some conflicts of interest between research group and external users, very low cash flow
1990-2000 Service unit	No paperwork for research group, well-organized structure, no conflicts of interest, high cash flow	Technical know-how of manager, problems with marketing
2000-2005 Scientific collaborations	No conflicts of interest, know-how in marketing and communications, high publication output, use of scientific network	Paperwork, need for discounts to universities, reduction of cash flow
2005–??? Business unit	?	?

3.1. NAA sample types

An overview of the sample types analyzed currently and 5 years ago is given in Table 2. Plastics have been a major market segment for long, being a perfect niche for INAA since it can be done non-destructive. The analyses dealt with the enforcement of the Cd decree for reduction of the use of Cd in plastics [8][9]. However, in 2001 also XRF was accepted as a screening technique upon extensive validation using INAA. This reduced the incoming stream of sample considerably. In addition, the forced change from industry oriented to academic oriented by about the year 2000 caused also a major change in the type of samples analyzed. By 2004, the majority of analyses were done for collaborative projects with epidemiologists in which nail clippings are involved. In addition, there is a small persisting market for analysis of new materials, composites, silicon carbide, carbon fiber, alumina etc. It should be noted that there is hardly an industrial market for geological and related analyses in the Netherlands. Moreover, the emphasis in environmental assessments is with water analysis and organic residues, whereas there is a remaining quest for determination of elements like Pb and Cd, for which NAA has none or very limited capabilities.

TABLE 2. PERCENTAGE INDICATIONS OF SAMPLE TYPES FOR INAA

Sample types	1995	2000	2005
Plastics	60	80	5
High-tech materials	5	5	5
Bio-indicators	20	10	10
Nail clippings	-	-	70
Sediments, soils, rocks	5	-	-
Others (e.g. catalysts)	5	5	10

The samples from internal users mostly result from various biomonitoring projects, for instance, lichens, mosses, tree bark, soil and air particulate matter.

3.2. NAA analysis protocols

The traditional protocol for multi-element analysis was and still is: 2 irradiations, 3 measurements (the shorts, one after 1 week and one measurement after about 1 month), 50–60 elements reporting. The various requests and particularly the needs of the external customers made necessary to develop different analysis protocols.

Most external customers appear not to be interested in full multi-element analysis, even not when the data is given for free together with the data requested in the first place. Customers are usually oriented to one or a few given elements (See Figure 1) whereas their main demand lies with turnaround time. Here lies a fundamental change to be made for an INAA laboratory when operating at the commercial market. "Good is good enough" implies that the analyst should refrain from traditional scientific protocols aiming at the best precision and detection limits. Many customers are interested in order of magnitude indications rather than indications with a precision, e.g., better than 10%.

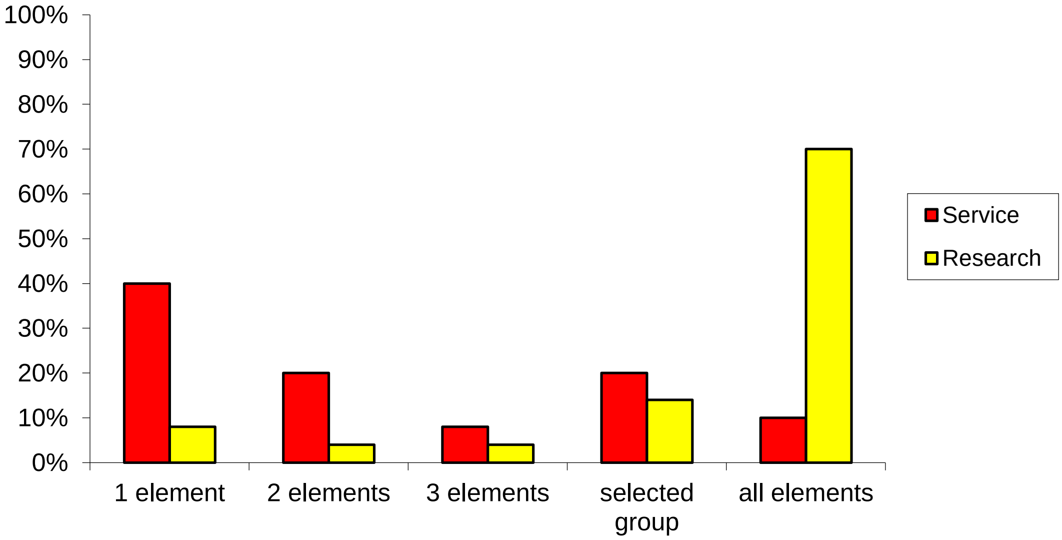

Fig. 1. Indication of interest in number of elements to be reported via INAA from industrial customers (service) and customers in scientific collaborations (research).

The majority of the work for external customers deals with one measurement 2–4 days after irradiation and determination of 1–10 elements. The turnaround time of these measurements is about 1 week–10 days (See Figure 2). This is usually acceptable for customers who compromise between number of elements and turnaround time. In addition, dedicated protocols have been developed too, in which routinely results could be reported within 3 working days turnaround time for a group of 12 elements.

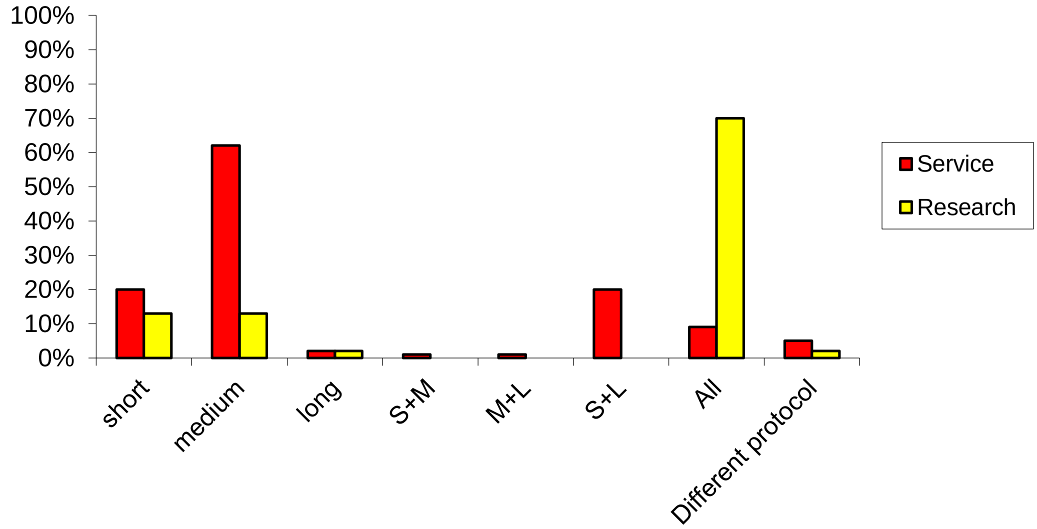

Fig. 2. Indication of analytical protocols, needed for samples from industrial customers (service) and customers in scientific collaborations (research). Typical conditions: Short: t_{irr}=5–30 s, t_d=0.5–10 m, t_c=0.5–5 m; Medium: t_{irr}=1–4 h, t_d=5 d, t_c=1 h; Long: t_{irr}=1–4 h, t_d=3 w, t_c=1–4 h.

The academic market segment, on the contrary, is mainly interested in full multi-element analyses at the best measurement capabilities. Experience with services to groups at other universities learned that requests for multi-element data changed rapidly in request for a limited number of elements when the groups were fully charged for the analyses.

3.3. Costs and tariffs

Cost analysis of INAA services in a university environment, and particularly in an institution operation a nuclear research reactor, is a complicated issue. Basically, the cost analysis is simple. Estimates are needed of the salaries of the people involved, the running costs of consumables in the laboratory, the depreciation of equipment and the costs of using the infrastructure. Problems started with e.g. estimation the costs of depreciation of equipment; the university prescribes 3 years whereas it is well-known that e.g. semiconductor detectors easily can last for 10 years or many more. Additionally, discussions came about the costs of equipment shared for research and services; equally so for consumables like liquid nitrogen. The costs of infrastructure, especially the costs of neutrons, was never formally decided upon at IRI, and actually never accounted for. In addition, it had to be decided if the number of samples, the number of spectra measured, the number of elements reported or another denominator should be taken to estimate the costs of the service.

The tariffs, however, should be also based on the market tariffs for analytical services as set by other analytical service laboratories. It should be noted that these organizations may not be striving to have each component of their services fully sustainable, as long as the overall organization remains sustainable.

Tuning the tariffs for INAA services to the market tariffs is needed to create a competitive position, especially in the staring phase. It is sometimes better to aim at building a network and trust as a reliable partner, even at prices under the cost level, than aiming at fully coverage by prices equal to the analysis costs.

4. NON-MEDICAL RADIOTRACER APPLICATIONS

Both INAA and the applications of the radiotracer method require custom tailored protocols. However, in INAA use is made of many standardized components such as irradiation, counting equipment and spectrum analysis software, whereas the radiotracer method is typically characterized by custom tailored experimental design, including source preparation and development, choice of equipment, collimator and data acquisition systems, up to modelling and tracer kinetic analysis. Radiotracer applications can therefore hardly be classified as routine, as they feasible for wide scale marketing.

There is a large potential of industrial applications in all applied sciences. In many cases the systems studied are remote, e.g., industrial installations or laboratories, environmental systems like estuaries or medical institutions. Radiotracer methods are characterized by non-invasive measurements which offer for industry the possibility of non-interrupted processes, a direct economic benefit. Unfortunately, these applications, with an outlook toward commercial services, are strongly hampered by requirements to licensing the use of radioactivity. Licensing procedures may be very time consuming irrespective of the risks involved, and authorities may sometimes formulate impractical or costly precautions. These licensing problems are one of the main reasons that most of the radiotracer applications by the Reactor Institute for third parties dealt with their use in scientific research on processes and systems of industrial relevance rather than in industrial problem-solving tasks [10].

Examples of radiotracer research of industrial relevancy are:

(i) **Classification of particles in a solid–liquid fluidized bed:** Fluidized beds are one of the most important chemical engineering technologies. This project aimed to estimate the velocity of a single particle falling down through a fluidized bed. To this end, ^{113m}In (half life: 99.5 m) labeled ion exchange particles were let to fall down a tube through a bed of smaller ion-exchanger particles fluidized by de-ionized water.

(ii) **Gas-liquid contacting in a stirred vessel:** Stirred vessels are also applied in chemical engineering to carry out reactions between gases and liquids. There is a continuous need to describe them in terms of basic engineering, physics and chemistry that underlie the fluid flow, interphase transfer and chemical reaction. Since argon matches oxygen in diffusivity and solubility in water, ^{41}Ar is an ideal tracer to study the oxygen residence time distribution in stirred vessels.

(iii) **Gas flow and mixing in a pressurized fluidized bed combustor:** A pressurized fluidized-bed combustor is one of the new technologies for environmental friendly burning of solid fossil fuel. The flow and mixing of gas have a significant influence on the heat transfer and the kinetics of the burning process. ^{41}Ar is a suitable tracer to determine the pattern of gas passage through the bed. New information was obtained that could be utilized for the development and design of industrial scale fluidized-bed coal combustors.

(iv) **Mixing of particulate solid in a batch-type solids mixer:** A Nauta mixer is a well-known and widely used industrial mixer for solids. The device consists of a conical hopper and a rotating and orbiting screw. Mixing times, the time needed to obtain an equilibrium mixture, were determined using ^{128}I labeled sand particles.

(v) **Movement of a textile piece in a laundry machine drum:** The influence of the mechanical agitation on the washing efficiency of a domestic drum-type fabric laundry machine is still subject of research. It is of course not easy to observe the individual movements of the textile and wash liquids. A solution was found by attaching a suitable radioactive source (^{99m}Tc) to a piece of textile and to visualize the position of that particular piece, as a function of time, using a gamma-camera. The project has led to improvements of both the laundry machine and the economics of the washing procedure.

The examples given above demonstrate that the radiotracer method offers a good outlook for services and creative solutions to industry. Some of the examples given were indirectly financed by the chemical industry, by making equipment available or via payment of the radiotracer production. The real cost effectiveness of such scientific services is low, since quite some effort is involved in the experimental design and data evaluation. The benefits for the mother organization are its embedding in the country's industrial infrastructure, with additional scientific spin-offs.

Once licensing has been realized, the production of radionuclides for industrial application of the radiotracer method can be economically attractive, especially for "specials" such as radionuclides with short half-lives, gaseous tracers or for custom tailored labeled compounds or particles. However, it is doubtful if the total turnover of such a service can grow to a same level as attainable by INAA.

5. MEASUREMENT OF NATURALLY OCCURRING RADIONUCLIDES

Measurement of naturally occurring radionuclides is a commercially trivial activity in many nuclear centres. The measurements mostly deal with determination of, e.g., ^{3}H, ^{137}Cs, or ^{90}Sr in food products or environmental systems. Laboratories performing gamma ray spectroscopy or liquid scintillation counting of these radionuclides can relatively easy be licensed which implies competition by outsiders.

Better opportunities for unique commercial services are with determination of alpha-radiation emitting radionuclides requiring radiochemical separations. Many sediments and ores are rich in uranium and thorium, and thus in the decay products of ^{238}U, ^{235}U and ^{232}Th. Phosphate ore, used for phosphoric acid, phosphate fertilizer and gypsum production, may serve as an example. The daughter isotopes of the uranium–radium series, particularly ^{226}Ra, ^{210}Po, ^{210}Pb, end-up in the gypsum fraction, and finally in the surface waters. The Reactor Institute in Delft was invited by such a phosphate fertilizer producing company to make an assessment of its discharges in view of renewal of its license. An estimate of the dose to the population was required which made necessary to determine the amounts of these radionuclides in the various steps in a production process. ^{226}Ra, ^{238}U, ^{235}U and ^{232}Th have been be determined by direct gamma ray spectroscopy, ^{210}Po and ^{210}Pb by radiochemical separations and alpha spectrometry and gamma ray spectroscopy, respectively.

A similar project on the determination of alpha emitting radionuclides and beta emitting radionuclides in Dutch freshwater systems was carried out for the Dutch Ministry of Environmental Affairs. Three kinds of alpha emitting and beta emitting radionuclides were distinguished:

- Natural sources of natural alpha and beta emitters;
- Human activities by which the levels of natural alpha or emitters is increased (‘technological enhancement”); and
- Nuclear facilities that enrich uranium, produce transuranic alpha emitters (power stations and other nuclear reactors), or produce anthropogenic beta emitters.

Characteristic for such projects is that the project teams are usually large; that extensive reporting is required but also that the assessments are not regularly returning, perhaps only once every 10 or more years. The position of the Nuclear Institute as a national expert centre for such assessments is, however, established.

Oil and natural gas exploration is another market for such assessments since it has been observed that radionuclides also accumulate in, e.g., ducts and at exploration rigs.

6. PITFALLS IN COMMERCIAL ACTIVITIES

The Reactor Institute has also been active in the color enhancement of topaz gem stones by reactor irradiation. In the 1980s the technology of changing the color of topaz stones from worthless colorless to more valuable blue was implemented at IRI and approaches were found to limit the induced radioactivity of impurities. Customers providing the colorless stones were attracted resulting in a large-scale activity. A dedicated irradiation facility was constructed as well as an automatic device in which, upon pre-defined cooling, the radioactivity of each individual stone could be assessed and the stones could be sorted as well. It all involved a considerable investment by the Institute, which was accounted for via the contract with the supplier. However, by the early 1990s the market for topaz gemstones had collapsed; the supplier went bankrupt and the Institute had to shut down its operations. In addition, the supplier had no funds any more to pay for the several kilograms of irradiated gemstones which remained, hitherto, stored in the Institute’s vaults.

Organizations seeing commercial marketing of their regular activities, as with nuclear techniques, should realize that it introduces also some vulnerability. Another example was already outlined in the above, for INAA of plastics. National regulations prescribed explicitly INAA as method of preference and for many years thousands of samples were analyzed annually by INAA for the Cd content. However, desktop XRF machines became available with satisfying performance for screening purposes, and this approach took largely away the market for INAA.

Another example deals with use of INAA for the determination of trace elements in soil using large sample INAA [11]. Here, though convinced of the benefits of large sample INAA, decision makers decided not to prescribe this method so as to avoid that the Reactor Institute would have a national monopoly position for such assays.

7. MARKETING, PUBLIC RELATIONS AND QUALITY ASPECTS

Scientists and especially university academics generally do not stand out by strictly keeping to deadlines. This is one of the main cultural changes, required for success in commercial activities. Time for reporting belong, together with price, to the first points of discussion in contacts with third parties.

Marketing of the business activities for INAA were already in an early stage (1980s) supported by the ‘contract bureau’ of Delft University. The first support dealt with advice on the preparation of hand-outs for use when operating a booth at fairs and exhibitions. Training in commercial application and approaching visitors at fairs also were taking into account. In the 1990s more professional help was provided in the preparation of brochures. This was eye-opening regarding the type and style of information in text and pictures to be displayed. This all was another cultural change: public information on INAA should contain not the scientific description and typical advantages of the techniques, but requires anticipation on the perception by potential clients. The photographs in the brochures were made by a professional in advertisement, who selected himself the photogenic parts of the laboratory, even if they were scientifically irrelevant.

Cost analysis has been a continuous point of discussion. The university developed general guidelines like hourly rates and depreciation rules for calculating the costs of its services. Several unresolved specific cases remained, such as the costs of equipment shared for research and for services; accounting for equipment that already passed the formal depreciation term; accounting for compensation ‘in natura’ by universities, e.g., via co-authorship of publications. The cost of the neutrons was never decided on by the institute’s management and left out from the calculations. In general, a certain degree of opportunism was often included in the price negotiations; also taking into account the tariffs set by other laboratories offering analytical services like AAS, ICP and XRF. The situation changed a little, as outlined in the above, when by the turn of the century the university accountants advised the Institute’s management to develop a tariff structure for INAA strictly applying the university rules. It would have raised the analysis price for third parties by 300%, which would completely put out of action. Therefore, it was decided to turn from pure commercial industry oriented services towards services in the frame of scientific collaborations.

The absence of a tariff structure for the use of the reactor neutrons also affects the cost estimation in radionuclide production. Projects, like the ones outlined above, were typically financed at mutual consent, largely covering either labor costs for production of radionuclides to be used by the customer himself or instrumentation costs when the application took place at the Institute.

Finally, a management infrastructure focused on quality appeared essential to reduce running costs by improving the efficiency and effectiveness of the operations. Implementation of a quality system, following the ISO/IEC 17025:1999, with associated accreditation, is now often inevitable [5] for testing laboratories to be competitive with other analytical laboratories offering routine services, whereas many ISO 9001 certified companies often only sub-contract analyses to a laboratory that is accredited. It can be shown that the additional costs of quality system implementation and accreditation are rapidly paid back from the reduction of costs of non-quality.

8. FINAL REMARKS

There are several opportunities to make nuclear analytical techniques available to society, and to demonstrate the socio-economic benefits. In fact, the various activities by the Radiochemistry department of the Reactor Institute in Delft were often embraced by the Institute’s management for public relation purposes and to improve the public view, e.g., of the citizens of Delft, on the viability of the nuclear institution and its research reactor.

There seems to be a worldwide tendency in universities to have regularly drastic changes of the organizational structure, of mission statements, policies and priority areas. Building confidence and credibility as a trustworthy partner in commercial communications may be strongly affected by this.

Radiotracers, especially those with half-lives in the order of hours to a few days, offer unique opportunities for field application and use on-site in the chemical industry, for example. The success of such applications depends entirely on the national regulations for licensing the use of radioactivity.

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