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History of inductively coupled plasma atomic emission spectral analysis: from the beginning up to its coupling with mass spectrometry

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An ionised and luminous gas is called a physical plasma. It contains free electrons and ions interacting with electric and magnetic fields. In the field of plasma physics this has been known for more than 150 years. Sometimes the physical plasma is called the fourth state of aggregation, because by supplying energy to the solid state, or aggregation, it normally passes at first into the liquid and thereafter into the gaseous state of aggregation. A further supply of energy will then lead to the physical plasma. Knowledge about physical plasma began with studies of gas discharge tubes by Julius Plücker (1801–1868) in Heidelberg and Bonn, and by his student Johann Wilhelm Hittorf (1824–1914), who later on examined electrodeless ring discharges in Münster (Germany). The two of them published an article in 1865 called, "On the spectra of ignited gases and vapours with regard to the same elementary gaseous substance".[1] Sir Joseph John

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I was born on January 30th 1934 in Berlin, Germany. From 1940 to1953 I attended elementary and high school, including the famous Waldoberschule Berlin-Charlottenburg from 1950. Then, from 1953 until 1960, I studied chemistry at the Technical University of Berlin and was a materials tester at SIEMENS cable factory in Berlin-Gartenfeld. Between 1960 and 1966 I was assistant to G. Jander and E. Asmus at the Chair of Analytical Chemistry, TU Berlin. In 1966 to the end of 1993 I was head of the chemical laboratories of HOESCH STAHLWERKE in Dortmund, the last 3 years I was Controller of the Research Department. Also, from 1985 up to 2001 I was a lecturer and honorary professor (1992) at Westphalia Wilhemns-University Münster. From 2001 to 2014 I was a scientific consultant for the SPETEC company in Erding.

I was born on January 22nd 1938 in Fraustadt, Germany (now Wschowa, Poland). From 1945 I attended elementary and high school in East-Germany, and also spent three years at a professional trade school to graduate as an electronic technician. In 1957 I escaped to West-Berlin and was employed by SIEMENS and attended evening classes at the GAUSS Engineering School. In 1959 I emigrated to the USA for 14 years, and was employed by Carl Zeiss, Inc. Sales-Service-Division in New York, where I was engaged with ophthalmology, electron microscopy, analytical chemistry and spectroscopy. I studied marketing at the University of Maryland and was a regional product specialist, based in Washington DC. In 1973 I returned to Germany as manager of materials science at KONTRON near Munich, this included atomic emission spectrometry, development project ICP source and sequential spectrometer. In 1980 to 2003 I worked as general manager of LECO Instrumente GmbH, a subsidiary of LECO Corp., St. Joseph, Mi, USA.

Thomson (1856–1940) explained the processes in plasmas[2] and discovered the electron. In 1928 the term "plasma" (from the Greek: $\pi\lambda\acute{\alpha}\sigma\mu\alpha$ = formation, structure) was introduced[3] by Irving Langmuir (1881–1951). Early efforts to maintain an induction plasma on a stream of gas could be attributed to G. I. Babat.[4] The 1960s were the initial period of thermal plasma technology. Thereafter, it was only a matter of time before analytical spectroscopists discovered induction plasma as a spectral excitation source.

The first application of such an induction plasma to the best of our knowledge was described by Eugen Bădărău (1887–1975) and co-workers, Bucharest (RO) about 60 years ago.[5]

At the VI Colloquium Spectroscopicum Internationale held in Amsterdam in 1956 Bădărău's team reported their studies about a high frequency torch discharge as a spectral source (Fig. 1). It looks like a single electrode induction plasma (later known as CMP). The nebulizer seems to have been the Babington type which was used later on for example as a Giess-McKinnen-Kaiser nebulizer. Unfortunately this publication was largely ignored, possibly because it was written in German. In the 1950s spectral analysis of solid conductive materials was already applied successfully for routine applications. Then the search started to analyse solutions in the same way. Further development took place in several directions. One of them (Fig. 2) was the deformation of the electric arc to a flame.[6] This approach resulted in the Plasma Jet[7] and ultimately in the burner of Erich Kranz (*1927). For the first time, the solution to be analysed was not in contact with the electrodes.[8]

Finally this research resulted in the DCP (Direct Current Plasma) approach.

1961–1965 was a period of great importance for the development of plasma excitation sources. It started in two separate paths which can be referred to as the English and the German-USA path.

The first approach led *via* G. I. Babat and T. B. Reed to Stanley Greenfield.[4] Reed used a high power induction torch source in 1961 to grow refractory

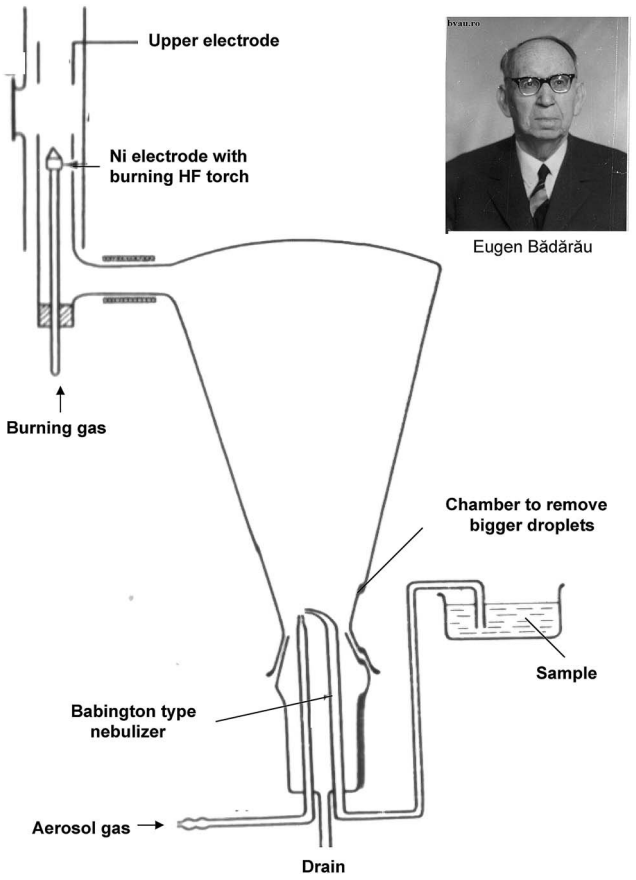

Fig. 1 First known plasma source.

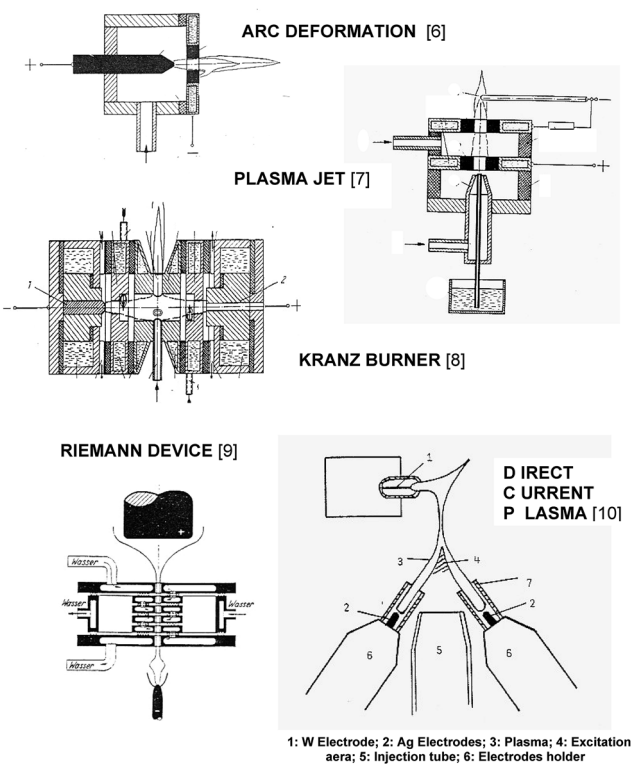

Fig. 2 Spectral analysis of solution.

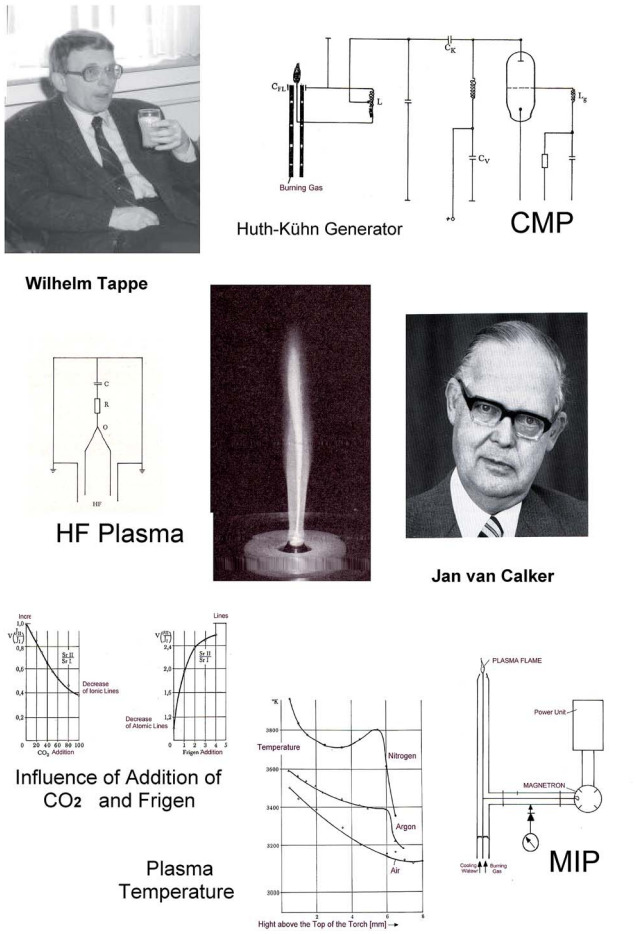

Fig. 3 Studies of Tappe at Münster.

crystals.[11] He was the first who protected the torch wall by a stream of argon.

The second approach was not as clearly structured, because several scientists initially investigated plasmas with different generators (direct, capacitive or inductively coupled). This took place almost simultaneously, resulting in the work of Velmer A. Fassel.

However, all of it actually began in Münster (Germany) where Jan van Calker (1918–1981) experimented with r.f. generators. Together with W. Tappe (*1934) he observed high current sparks and tested their use as a source within his thesis.[12] He also compared microwave induced plasma with capacitive coupled microwave plasma and inductively coupled plasma excitation.[13,14] He tested the appropriate generators, searched for the best usable frequency, and applied different burning gases (argon, nitrogen and air). Spectra were recorded and detection limits were obtained spectrographically by using photographic plates (in μg ml^{-1} level).

In the same year (1963) also U. Jecht and W. Kessler from Munich studied various plasma sources (MIP) for analytical application.[15]

In Japan, M. Yamamoto and S. Murayama described the electrode erosion in a radio-frequency torch discharge,[16] possibly to use the metal vapour for coating procedures.

In 1964 Tappe visited the USA and reported his results (Fig. 3).

In the meantime; R. Mavrodineanu and R. C. Hughes had examined all possible plasma sources including a low power one at 30 MHz and the MIP at 2.4 GHz.[17] One year later C. D. West and D. N. Hume had built their spectrometer, which used plasma excitation mainly in the MIP mode.[18]

At the same time H. Dunken, G. Pforr and W. Mikkeleit were operating an ultrasonic nebulizer to insert analyte solutions into a plasma excitation source[19] in Jena (Germany).

By 1964 the first route of development was successfully completed.

S. Greenfield was the first to apply an inductively coupled plasma source generated by a high power generator for analytical purposes at the company Albright & Wilson in the UK.[20] Greenfield examined different possibilities in his laboratory; inserting the analytical solutions by means of the injection technique in small volumes. Hence, his ICP was called the Greenfield plasma which meant high power of more than 3 kW with nitrogen as a cooling gas and argon as plasma gas (Fig. 4). He once said: "You can put anything you want into the plasma, it still works". In any case there was also enough time for tuning and the practical application of his ICP would be the only example for more than 10 years.

It is typical that developments in industry take a long time before they are accepted by research laboratories, universities or manufacturers.

One exception in this respect was V. A. Fassel (1919–1998), the former director of the Ames Laboratory at Iowa State University and the US Atomic Commission, later called Energy and Mineral Resources Research Institute. After W. H. Tappe visited Fassels laboratory the developmental process started to move quickly. Thus, in 1965 appeared the first publication on the use of plasma spectral excitation by R. H. Wendt and Fassel[21] which was quickly followed by others[23,27,31] for instance by Greenfield and co-workers[20,22,40,42] in the following years. The ICP described by Fassel and coworkers was of low power (about 1–2 kW) using argon as a cooling gas as well as plasma gas, and it was called the Fassel plasma.

In the second half of the sixties research increasingly dealt with the principles of ultrasonic nebulization,[24] and general applications of ICP. This work was also performed and reported by state institutions in London[25] and Rome.[26] The beginning of ICP research in France was started by J. Robin and

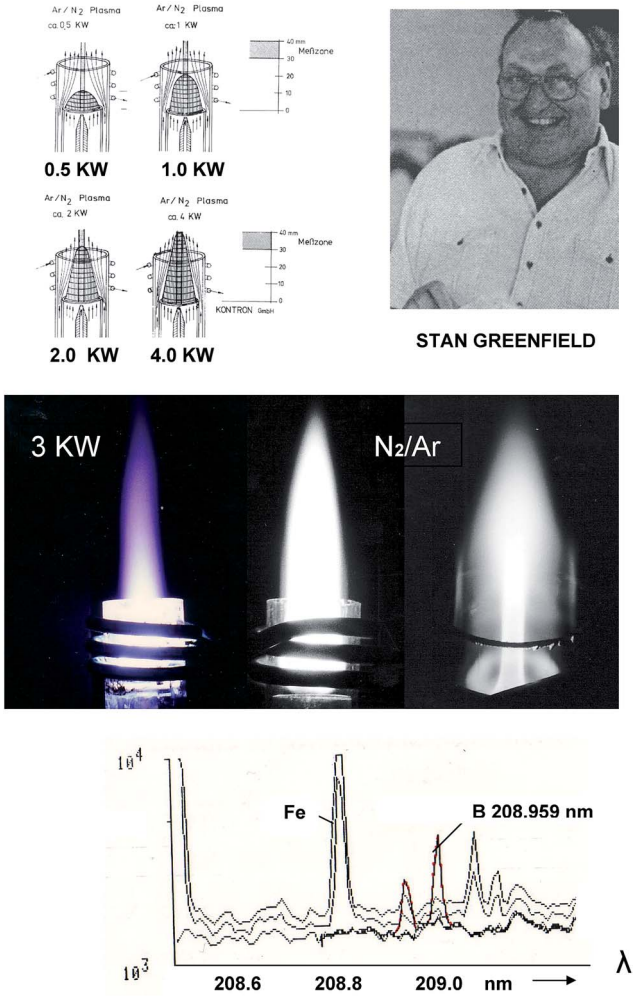

Fig. 4 The Greenfield plasma.

J. Michel Mermet using ultrasonic nebulization in Lyon,[28] where J. van Calker gave his lecture at the VI C.S.I. in 1961. M. Margoshes also started the evaluation of ICP[29] and the Fassel team could already determine elements at the nanogram per millilitre level.[31] In addition to the theoretical studies of L. de Galan, R. Smith and J. D. Wine-fordner[30] or R. C. Miller and R. J. Ayen,[34] one should also remember the early studies by the group of I. Kleinmann[32,38] in Prague (CZ) and the private investigations of E. Kranz[33] in Ilmenau (East-Germany). Following the reform of universities in 1968 all research on plasma spectroscopy was cancelled in East-Germany. At that time no purchasable spectrometer working with ICP excitation was available world-wide.

By the early seventies the long awaited first ICP instruments were supplied. Comparison with MIP excitation was important to realize the benefits of ICP,[35] which was also mentioned in a handbook.[36] Primarily due to the work of Greenfield and co-workers,[39,40,42] new work was published including the first publication in Japan,[37] G. F. Kirkbright in London[41] and J. Robin in Lyon.[43] They were studying optimized conditions for the ICP[38] and methods for the determination of elements in different sample materials. For the first time, a publication by P. Boumans and a co-worker from Eindhoven appeared.[44] Both were working for the Philips company which was a potential manufacturer of optical emission spectrometer devices at the time.

1973 and 1974 became very important for the further development of ICP excitation, instrumentation and analytical methods. Both authors of this Editorial met R. M. Barnes and V. A. Fassel at the XVII C.S.I. 1973 in Florence after Barnes' lecture. Our meeting with the two scientists from the USA was of an unusual mixture because we were from a commercial laboratory (HOESCH Steel Company) and an analytical instrument sales engineering company (KONTRON), and to say the least, we were inspired by their work.

To further explain, KONTRON had entered the atomic emission spectroscopy business in Europe with a USA manufactured model LABTEST V 25 in early 1973. Therefore, the 1973 Colloquium Spectroscopicum Internationale held in Florence (Italy) for KONTRON was an ideal forum to meet many leading analytical chemists and spectroscopy users in one location. An invitation dinner in the hotel Villa Medici had been organized by KONTRON to bring the attendees together (110 by number) in a casual atmosphere.

From this conference and dinner party a group of laboratory leaders, primarily from the German steel companies, evolved taking a closer interest in the LABTEST technology. This in turn led to a product conference in Munich, the home of KONTRON, in February 1974.

In the course of conversation on the subject of "ICP", the work of Fassel and Barnes was brought up by the laboratory chief of HOESCH and was picked up favourably by the manager of the new department at KONTRON who happened to be sitting at the same table. What followed was a visit by the two, now friends and authors, to the institute of V. A. Fassel at Iowa State University in Ames and the laboratory of R. M. Barnes at the University of Massachusetts in Amherst only one month later. It was the start of successful cooperation between an industrial laboratory and an engineering-marketing company. Some of the main results are described in this paper.

While in the previous 3 years, a total of only 9 papers on ICP were published, the number thereafter increased to twelve. The working groups of the Fassel

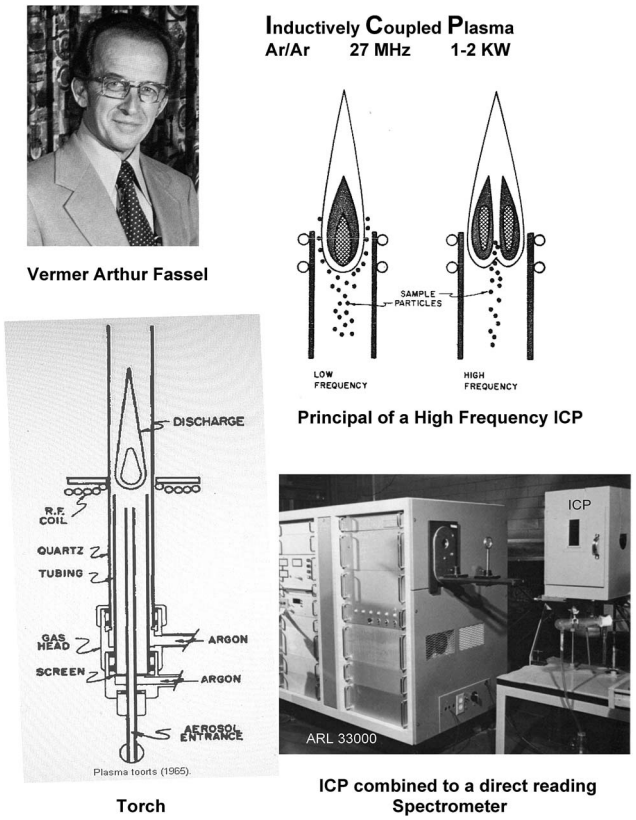

Fig. 5 The Fassel plasma.

team at Iowa were engaged in the determination of trace metals in micro litre volumes,[47] and gave a summary of current knowledge[53] and improved the nebulization of samples.[54] For the first time the evaporation of micro litre volumes from a tantalum filament was described.[52] In Lyon (F) J. Robin and J. M. Mermet were concerned with fundamental questions[49,55] and had already mixed gas plasmas.[50] In addition, the working group of L. de Galan started in Delft (NL) with baseline studies[56] and the team of G. F. Kirkbright in London (GB) determined some elements by using the ICP excitation[46] and studied a nitrogen–acetylene plasma source,[57] while R. H. Scott from Pretoria working in the Fassel laboratory in Ames was concerned with trace analysis of solutions.[51] According to Boumans from Philips, the Englishman M. Sermin presented the first results of Applied Research Laboratories (ARL) in Ecublens (CH) as the second manufacturer of instrumentation.[48] The application of an ARL spectrometer to an ICP excitation took place in the Ames Laboratory (Fig. 5).

In April 1974 the two authors of this paper visited Barnes at the University of Massachusetts in Amherst, and Fassel and co-workers at his Ames Laboratory to learn about the current state of practical instrumentation of the ICP as an analytical excitation source.

Right then and there the authors decided to engage in this technology. As a consequence of it a visit to company HENRY RADIO in Los Angeles followed where a 2 kW r.f. generator was purchased by HOESCH, the same as was used in Fassel's Ames Laboratory.

Back at home we started immediately with the physical construction of all supporting devices to conduct an analysis once the generator arrived and became integrated. KONTRON provided the mechanical electrical-gas supply base and the LABTEST V 25 atomic emission spectrometer, distributed by KONTRON at that time. The glassblower of the HOESCH research department produced the torch system along with nebulization chamber according to specifications set forth by Fassel and Scott. The workshop built an x, y, z-table for the matchbox, torch and chamber system.

By the summer of 1974 (Fig. 6) the first Fassel plasma was running for the first time in an industrial laboratory and the first elements contained in steel and oxides could be determined. This event took place ten years after the practical application of an ICP-AES by Greenfield in England, so it was about time for another user-application to be born.

Since the HENRY generator was produced in 1975 exclusively for the ARL manufacturer, KONTRON had to look for another company who could construct and build a suitable generator in Europe. LINN High Therm was found in Bavaria.

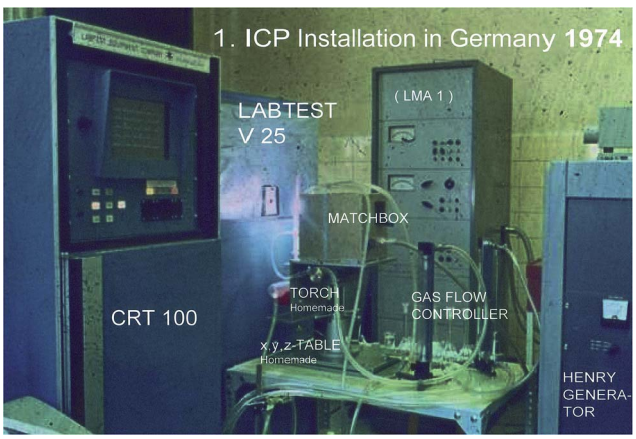

Fig. 6 ICP installation in Germany.

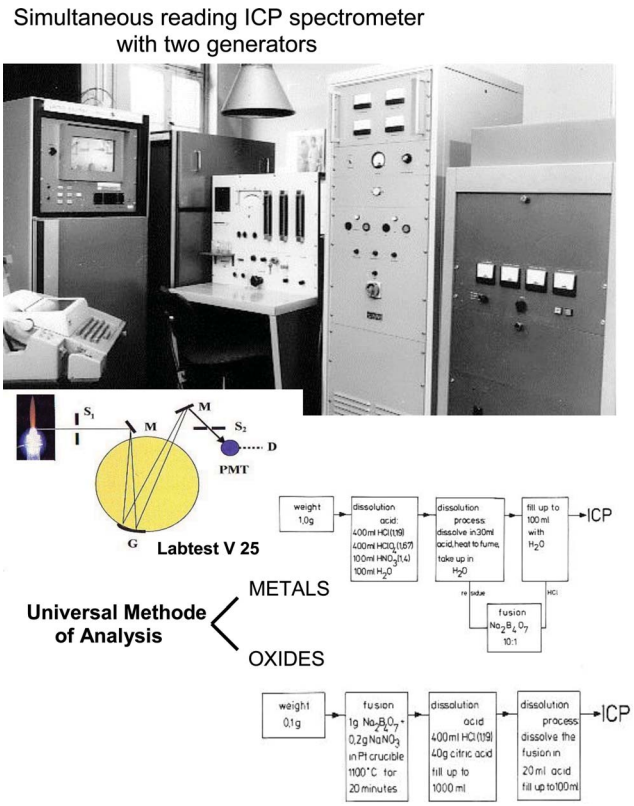

Fig. 7 Simultaneous element readout spectrometer system with 2 generators.

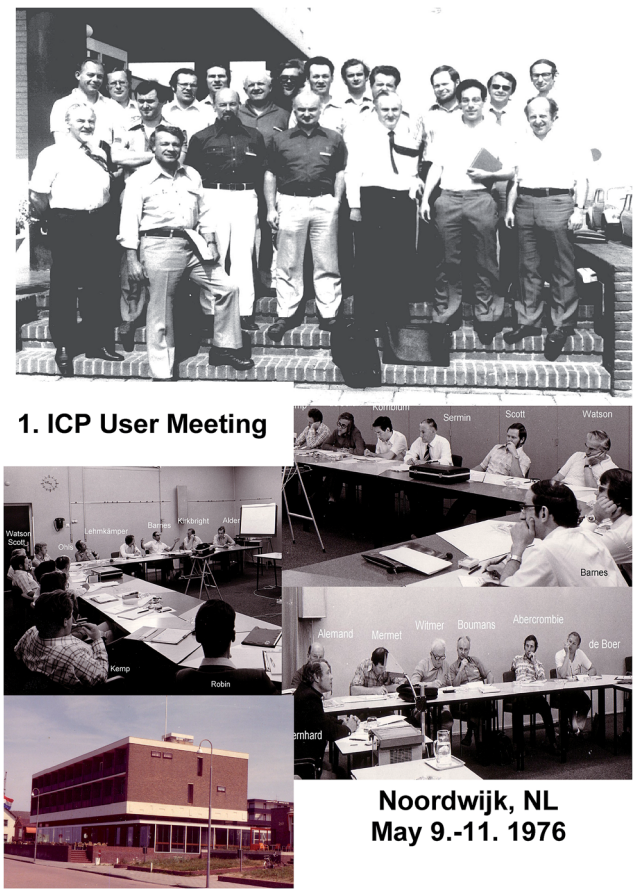

Fig. 8 User meeting in Noordwijk.

At that time LINN was already a manufacturer and supplier of high frequency induction metal remelt and oxidic fusion systems for KONTRON.

LINN High Therm was able and ready to develop and build a free-running generator in which the frequency slightly changed when the conductivity in the plasma oscillated, but not the performance. This changed in the crystal stabilized generator of HENRY in which the frequency remained stable, so we were able to test both Fassel and Greenfield plasma techniques.

In the meantime KONTRON had also optimised the area of matchbox, gas supply and burner system (Fig. 7). The uniqueness of the LABTEST V 25 was the simultaneous element readout system CRT 100. It enabled the user to observe the sensitivity of each element during analysis. This was helpful because the optimized selection of element lines for every matrix was not known at the time.

The first instrument for ICP spectral analysis was produced in Germany by KONTRON about 1 year later. It was exhibited at the XVII Colloquium Spectroscopicum Internationale 1975 held in Grenoble (F). However, only some experts like Robin and Boumans were interested in the new instrumental construction at that time. This participation also meant that we were invited for the 1 ICP User Meeting which was held in Noordwijk (NL) in May 1976. At the invitation of Leo de Galan, 20 participants from around the world (Fig. 8) discussed the progress of ICP for many hours.

Participants in the group picture (from left to right) were: Watson (Randsburg, RSA), de Boer (Eindhoven, NL), Kirkbright (London, GB), Mermet (Lyon, F), Bernhard (LosAngeles, USA), Demers (Cambridge, USA), Abercrombie (Ottawa, CDN), Alemand (Boston, USA), Witmer (Eindhoven, NL) de Galan (Delft, NL), Boumans (Eindhoven, NL), Robin (Lyon, F), Alder (London, GB), Kemp (Brussels B), Sermin (Ecublens, CH), Scott (Pretoria, RSA) Kornblum (Delft, NL), Lehmkämper (KONTRON Munich, D), Ohls (HOESCH Dortmund, D) and Barnes (Amherst, USA). Ten representatives came from universities and research institutes, 9 were employees of

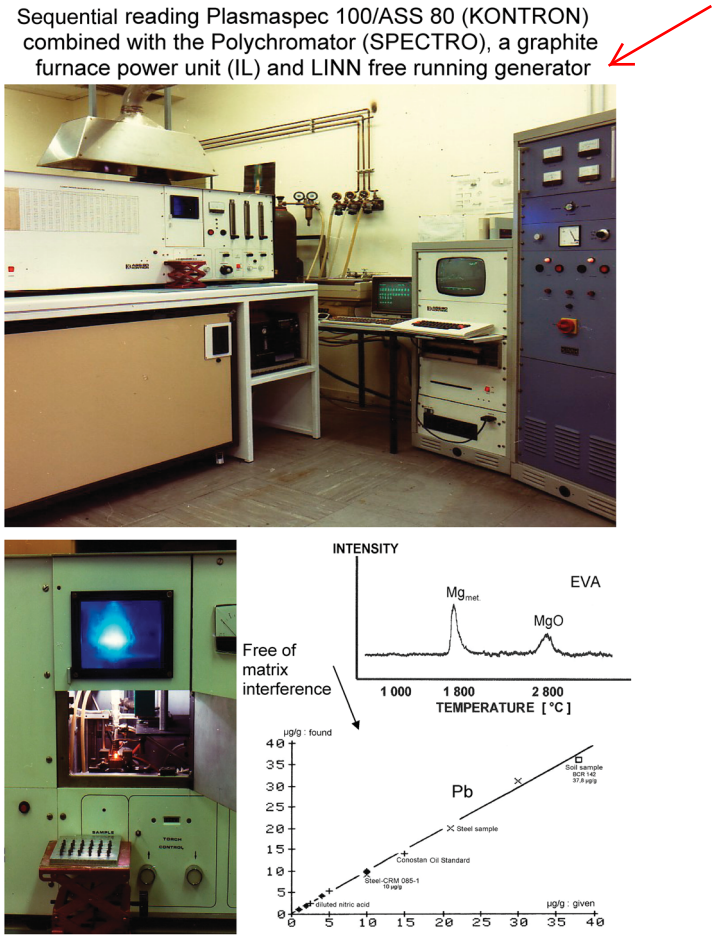

Fig. 9 KONTRON Plasmaspec/ASS 80 and SPECTRO Polyvac.

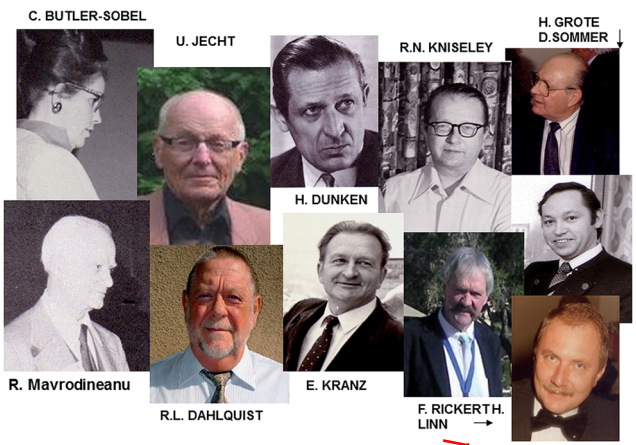

Fig. 10 ICP pioneers.

instrument manufacturers and only 1 was from an industrial laboratory.

Following this meeting most participants travelled to Munich (D), to attend the 1 European ICP Symposium organized by KONTRON. Greenfield also joined at this point and reported his results.

Twenty-four scientific papers were published in 1975 (13) and 1976 (11) from the already known groups of Fassel,[58,59,68,71,78,79] Barnes,[60,64,77] Greenfield,[61,72,81] Robin/Mermet,[62,63,66,73,74] Boumans,[65,67,80] Scott[69,70,75] and Horlick.[76]

Thereafter instrumentation development at KONTRON and ARL took the lead, followed by PHILIPS, BAIRD ATOMIC, JARELL-ASH, LABTEST, INSTRUMENTATION LABORATORY (IL) and SPECTRO.

Considering the example of ICP instrumentation and application development, we see that from now on spectrometer manufacturers had almost completely taken over the practical analysis development, because the benefits of this technology had become obvious to manufacturers and users alike.

KONTRON developed a sequential measuring spectrometer as well, called Plasmaspec 100, linked to the data processing system ASS 80 and an optical encoder-scanner for rapid wavelength positioning. Our early experience with the simultaneous reading spectrometer, using fixed wavelength positions, did show that not all analysis lines were properly set and that a sequential monochromator driven optical system would be useful. Meanwhile, ICP had replaced atomic absorption spectrometry sample analysis tasks in some laboratories, especially where several elements had to be determined simultaneously.

When Leo de Galan was invited to Noordwijk again after two years (1978), we had already installed two universal method to analyse metals and oxides. This second user meeting took place with almost all the same participants as at the first one.

Barnes founded a conference on plasma spectrochemistry in a two year sequence starting in 1980. He also had founded the ICP Information Newsletter in 1975 which has reported any plasma activity from around the world for the past 41 years.

Between 1977 and 1979 the number of publications on ICP applications remained relatively constant (13; 11; 14). In addition to the previously known groups of Fassel,[83,112-114,116] Mermet,[84,85,87,88,102,103] de Galan,[89,91] Boumans,[90,117] Greenfield,[95] Horlick,[99,110] Barnes[101,105] and Scott,[104] other groups came into the field namely: in 1977, Denton,[82] Yoshida[86] and Ohls;[92-94,106,107] in 1978 work was reported by Kirkbright,[96,97,111] Dahlquist[98] and Butler-Sobel;[100] then in 1979 Hieftje,[108]

Omenetto,[109] Demers,[115] Broekaert[118] and Janssens[119] published papers for the first time.

In an industrial laboratory it was possible to combine equipment parts from different manufacturers, such as the KONTRON Plasmaspec/ASS 80 with the three polychromator instrument of SPECTRO and the graphite furnace unit of INSTRUMENTATION LABORATORY (Fig. 9). This combination created opportunities for direct solid sample analysis, *e.g.*, the species determination of magnesium in steel, the injection technique of microliter sample volumes, and the use as a detector for gas chromatographic analysis.

Very important for further development was the first Winter Conference on Plasma Spectrochemistry held in San Juan, Puerto Rico in January 1980. This conference organised by Barnes[124] is still running every two years, and there is now also the European Winter Conference on Plasma Spectrochemistry which has been organized by different scientists from various countries every two years since 1985. The increase of published ICP papers after 1980 did show the progress of application and the first line tables published[121-123] and manuals summarized previous results in the following years.[125-127]

Finally, the initial development of spectrometry using plasma excitation would not be complete without mentioning the pioneers (Fig. 10), who have not previously featured in this paper.

Since 1980 ICP atomic emission spectrometry has been used worldwide, but the instrumental technique has not been methodically improved until the following few years. Academic interest turned to the new ICP technique in 1980; the combination of ICP with mass spectrometric detection.[120]

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