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Fifty years since the first European synchrotron-radiation-derived XAFS spectrum (Frascati, 1963)

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The first absorption spectra recorded in Europe using synchrotron radiation as the X-ray source were the *K*-edge of Al and the L_{III}-edge of Cu taken at Frascati electron synchrotron in May 1963 by the French–Italian group comprised of Y. Cauchois, C. Bonnelle and G. Missoni.

Keywords: electron synchrotron; absorption; emission; *K*-edge; *L*-edge; Al; Cu.

1. The Italian synchrotron facility at Frascati

There has been some controversy among science historians over who first predicted and first detected synchrotron radiation. According to some, the first theoretical demonstration that electrons orbiting within a magnetic field emit photons arose in the minds of the Russian physicists D. D. Ivanenko and I. Ya. Pomeranchuk in 1944; according to others, the first scientist who published the theory was V. I. Veksler, another Russian physicist and in the same year. Similarly, E. M. McMillan is credited with having built the first electron synchrotron at Berkeley, CA, USA, in 1945, and naming this new type of particle accelerator; yet, the first calculation of the energy loss of charged particles in circular motion in the USA is by J. P. Blewett, who was studying the 100 MeV betatron of General Electric at Schenectady, NY. Indeed, the first observation of a bright arc of light (*i.e.* of visible synchrotron radiation) was made at Schenectady, in 1947, by a General Electric technician attending the accelerator machine that H. C. Pollock was refurbishing to transform it into a 70 MeV electron synchrotron. This years-long controversy is well known and documented (Pollock, 1983). There is also some controversy concerning the first synchrotron to operate in Europe: there may have been one located somewhere in Moscow, USSR, but the information on it is practically non-existent because it fell under the closely guarded secrets of the Cold War; on the other hand, it is certain that the first electron synchrotron to operate in Western Europe was that at the Frascati laboratories near Rome, Italy (Haensel, 1994, p. 15).

1.1. Establishing the Italian synchrotron

On 19 January 1953, Gilberto Bernardini presented a proposal to build an accelerator of power 500–1000 MeV in Italy. The first funding came from a portion of the yearly grant received by the Istituto Nazionale di Fisica Nucleare (INFN), at that time a division of Consiglio Nazionale delle Ricerche (CNR), an Italian government institution supported by public funds. However, very soon most expenses were taken over by Comitato Nazionale delle Ricerche Nucleari (CNRN), which then, in an age of enthusiasm for atomic energy, enjoyed much larger support from the state. The project was under the direction of Giorgio Salvini, who took upon himself the responsibility of building an electron synchrotron instead of a linear accelerator. The town of Frascati, located some 20 km southeast of Rome, was chosen to host the machine. It took four years to prepare the laboratory hall at Frascati, while the design of the apparatus started in the summer of 1953 at the Institute of Physics of Pisa University. Later, in May 1955, the team of the Sezione Acceleratore moved from Pisa to Rome University to better follow the work in progress. In the summer of 1957, when a truck travelled ten times back and forth from Pisa to Frascati to transport the already built components, the project was at its final stage. This is also the year when the Laboratori Nazionali del Sincrotrone (LNF) of Istituto Nazionale di Fisica Nucleare (INFN) was officially born. Finally, in 1958, the assembly and the commissioning of the synchrotron started. The Frascati electron synchrotron reached full operation at 1000 MeV on 9 February 1959, the foreseen further target being to reach 1100–1200 MeV (Salvini, 1962[1]; see also Salvini, 2008).

Congruently with the INFN *raison d'être*, the electron synchrotron (Fig. 1) was primarily dedicated to research in nuclear physics, such as pion–nucleon resonance, electron–pion diffusion, neutral pion decay and the properties of the η meson. Indeed, the first users had nothing else in mind but nuclear physics, and they concentrated all their experimental skills on this field. Their results were interesting and widely appreciated.

During operation, an electron synchrotron incoherently emits photons as the circulating electrons slow down. The photons are within a quasi-monochromatic, homogeneous and $\sim$70% polarized beam of high resolution ($>10^{-3}$) when interacting with a single crystal of diamond (Missoni & Ruggiero, 1965; see also Barbiellini *et al.*, 1962*a*,*b*). The availability of a beam of photons having a spectral distribution extending continuously from the microwave to the soft X-ray region in spite of the rather weak intensity (Balzarotti *et al.*, 1970) should have offered Italian physicists the possibility of entering a field of research they had never approached before, *i.e.* X-ray spectroscopy. They did not. Nonetheless, this missed opportunity turned out to be a profit for science, because it generated an

[1] The unpublished report ISS 62/7 dated 1 March 1962, prepared by M. Ageno and G. Missoni, clarifies (pp. 64–66) that the task assigned to the ISS group was to extract radiation from the synchrotron doughnut to test it and to make it available for a variety of physics experiments including biophysics; research on spectrography was being performed in cooperation with the French group, which had taken responsibility for setting up an appropriate spectrograph for spectral analysis of soft X-rays and the production of the monochromatic beam.

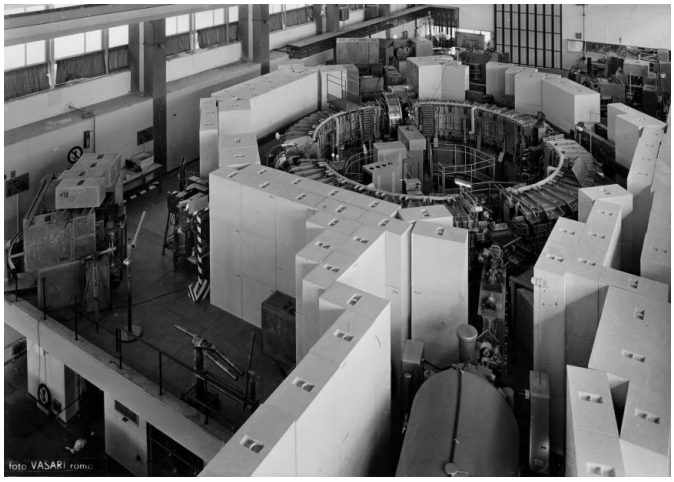


Figure 1
View of the Frascati electron synchrotron, in operation from 1959 to 1975, with all the beamlines derived from it. The Italian–French cooperative experimental set-up was located in the front left-hand side sector.

unprecedented international collaboration in Europe that paved the way to establishing CERN, the most ambitious centre for 'big science' worldwide.

1.2. Yvette Cauchois' contribution to X-ray absorption spectroscopy

Yvette Cauchois (1908–1999) was one of the very few women of her time to be admitted at a university in France and to graduate in physics (1928). Then she attended the Sorbonne University of Paris and earned a Doctorat de troisième cycle (1933) under Jean Perrin, the Nobel Laureate in Physics in 1926. She dedicated all her life to physics. In fact, her area in physics was X-ray spectroscopy, a rather restricted field at that time, which, however, had new, illustrious tradition in France and is now identified as X-ray absorption spectroscopy (XAS). Indeed, Maurice de Broglie (de Broglie, 1913*a*), concurrently with Julius Herweg in Germany (Herweg, 1913)[2], was the first to detect and consider the series of dark and light bands, which follow the absorption edge of an element impinged by X-rays; he called the bands a spectrum, and XAS the branch of science studying the properties of such a spectrum. His commitment to X-ray absorption was in clear contrast to that of the many scientists then studying X-rays under the viewpoints of emission and diffraction, and developing the interaction between these phenomena and matter as the leading branches of quantum physics. In the long run, X-ray diffraction (XRD) studies progressed so much on ordered solid matter, *i.e.* crystalline chemical systems, as to start being comprehensively called crystallography, mostly owing the name to the diffraction images produced from natural and synthetic crystals when exposed to the characteristic X-ray radiation beam emitted by a discharge tube. Consequently, in France, Maurice de Broglie was almost alone in his effort to promote XAS during 1910–1920, when most if not all 'big science' was concentrated on XRD, as it was elsewhere (*e.g.* M. von Laue, W. L. Bragg, P. Debye, P. P. Ewald, *etc.*). Actually XAS was studied as a second-rank field. The best qualified researchers in this field were M. Siegbahn, D. Coster, R. Kronig and W. Kossel. The first Nobel Prize in physics '*for his discoveries and research in the field of X-ray spectroscopy*' was awarded to M. Siegbahn in 1924, ten years after the first prize in physics in the field of X-ray diffraction (1914, M. Laue), which had been closely followed by that awarded to W. H. and W. L. Bragg in 1915.

[2] Actually, the discovery of the absorption spectrum is another matter of historical controversy: Herweg may have preceded de Broglie by a few months, judging by the date of presentation of their papers (30 June *versus* 22 December 1913), but he gave up after the first unfavourable remark, whereas de Broglie persisted and perfected his work. However, quite likely, the first recorded 'fine structure' is due to W. Stenström, in 1918.

Figure 2
Yvette Cauchois (1908–1999). Photograph taken in 1960.

In France, Yvette Cauchois' decision to dedicate herself almost entirely to XAS did not receive opposition. She was left free to work, but was mostly alone with little support. During the 1930s she developed bent-crystal spectrographs, determined numerous emission and absorption lines, and established reliable values for the binding energies of many elements. After World War II, having been granted a chair at Sorbonne University (Fig. 2), she took up writing books for the education of young physicists and published her experimental results. Her most remarkable contribution was a book in the form of tables (Cauchois & Holubei, 1947) that rapidly became an internationally recognized reference. Moreover, as a rather rare case among full professors at the Paris University, she did not give up personal research entirely, and stimulated students and researchers to go on with X-ray research, always pushing them to look for new innovative aspects of an apparently endless open field. Cauchois' merits and achievements are extensively described elsewhere (Wuilleumier, 2003).

1.3. Beginning the French–Italian cooperation on synchrotron

As the Director of the Laboratory of Physical Chemistry of the Faculty of Science at Paris University, in 1961 Yvette Cauchois had the brilliant idea of starting a cooperation with Mario Ageno (Fig. 3), at that time the Director of the Laboratory of Physics at the Istituto Superiore di Sanità (ISS) in Rome. Their joint program mainly concerned the determination of the photoionization cross sections of elements with high atomic number, at that time possible only through the intense emission of the Frascati electron synchrotron. Ageno and his co-workers had contributed in setting it up by designing and building a Cockcroft–Walton type of injector (*cf.* Salvini, 1962; see also Salvini, 2008); therefore, they had access to the synchrotron, although with some limitations because, at the suggestion of Enrico Fermi, the final injector used was not theirs, but a more powerful commercial Van de Graaff one. After a delay of more than one year, used to set up a grating spectrograph in Paris (Jaeglé, 1966) of dimensions and stability that would fit the space made available to them by the new Director of the facility, Italo Federico Quercia, in April 1963 Cauchois, together with a group of students, researchers and technicians, moved to Frascati with her apparatus, set everything

Figure 3
Mario Ageno (1915–1992) at the electron microscope of Istituto Superiore di Sanità, Roma, *ca* 1960. Courtesy of 'Archivio storico fotografico' of Settore Attività Editoriali dell'Istituto Superiore di Sanità, Roma, with permission granted for publication.

up next to the synchrotron doughnut (Fig. 1) and started making good use of the limited beam time allotted to her. Her first paper (Cauchois *et al.*, 1963*a*), written in cooperation with her pupil Christiane Bonnelle and with Guido Missoni, a researcher at ISS whom Ageno had appointed to the job, describes carefully the entire set-up and provides the first results, but tells us little about the difficulties encountered. They are described vividly by Pierre Jaeglé, her main co-worker, in a recollection written 25 years later, describing the available space as '*scarcely more than three cubic meters, separated from the huge synchrotron hall by a large, heavy, fireproof, black-plastic sheet – that was our soft X-ray laboratory. Inside, 40° Celsius. Our apparatus ... often had to be moved during maintenance operation. The only fixed point and the only guarantee that the laboratory could recover its place was the valve at the end of an araldite pipe*' (Jaeglé, 1989, p. 22)[3].

2. Experimental results

2.1. Absorption spectra

The first scientific information gathered by Cauchois and co-workers in their pioneering studies conducted at Frascati electron synchrotron was twofold: (*a*) the radiation emitted at 1 GeV covered the electromagnetic spectrum from the X-ray range to visible, with maximum intensity at about 10 Å (*i.e.* ∼1240 eV), thus interesting for their aims ['*interéssante*' (Cauchois *et al.*, 1963*a*, p. 409)] when compared with the bremsstrahlung emitted by conventional sources; indeed, it was particularly suited to the biological research (Jaeglé, 1966, p. 409) they intended to carry out; (*b*) the use of the bent-crystal spectrograph coupled with photographic recording was possible despite the small space allotted (Cauchois *et al.*, 1963*a*, p. 409) and the instability of the synchrotron beam. The instrument Cauchois had installed at Frascati (Jaeglé, 1966, 1989; Cauchois *et al.*, 1963*a*) operated under vacuum in the 2–25 Å range (∼6200–500 eV) and had a bent-crystal monochromator that could be rapidly replaced during the run: quartz, gypsum and micas could be exchanged according to the needs. The first results, on the other hand, were very exciting (Cauchois *et al.*, 1963*a*, p. 412), and not so much for the accuracy of the measured absorption edges [Al-K = 1563 eV and Cu-L_{III} = 932 eV[4], with the former edge showing up only as a deep discontinuity in the overexposed plate (Fig. 4*a*), and the second one being instead a clear jump followed by a fine structure (Fig. 4*b*)]; what appeared to be most interesting was the significantly shorter exposure time, which turned out to be of the order of '*quelques dizaines*' (Cauchois *et al.*, 1963*a*, p. 402), up to 50 times (Cauchois *et al.*, 1963*b*, p. 1243) shorter than what could be expected from the best conventional discharge tube.

Moreover, Cauchois *et al.* (1963*a*, p. 402) could proudly boast that their results were the first obtained at relatively high energies and by using a crystal analyser, whereas the only two previously published XAS results using synchrotron radiation [the Be K-edge at 112 eV and the Al L_{III}-edge at 72.8 eV (Tomboulian & Hartman, 1956), and the absorptions of gases in the energy range 26–69 eV (Madden & Codling, 1963)] that they were aware of, both obtained in the USA, were at much lower energy. Surprisingly, Cauchois disregarded, or was unaware of, the very early experiment conducted by Johnston & Tomboulian in 1954 (Johnston & Tomboulian, 1954). Indeed, this extraordinary precocious experiment had given to the physics community the first demonstration of an XAS spectrum recorded from polychromatic synchrotron radiation. Moreover, she did not refer to the theoretical paper where Parrat (1959) had mathematically shown the advantage of using synchrotron radiation for XAS instead of conventional X-ray sources.

2.2. Emission results

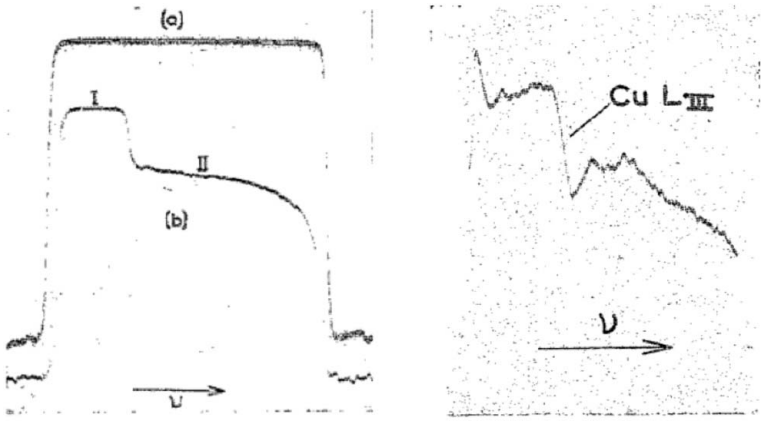
To conclude, a short addition is appropriate, although it is off the main topic. In their second paper, Cauchois *et al.* (1963*b* p. 1243) took advantage of the strong radiation that the Frascati synchrotron emitted when operating at 1.1 GeV (and, in addition, of the flexibility of their spectrometer) to measure the fluorescence emission line of Al. In 20 min and by using a gypsum crystal they could register the entire $K\alpha$ emission line at 1493 eV and, after exchanging the analyser crystal with quartz, they could even notice that after just a 2.5 min exposure the Al-K line splits into the doublet $K\alpha_1,\alpha_2$ (Fig. 5).

3. International scientific development

Quite satisfied with the results obtained, the French scientists over the years kept visiting Frascati several times (Jaeglé & Missoni, 1966; Jaeglé *et al.*, 1967, 1968*a*,*b*), conducting experiments, mostly with Ageno's research group, that focused on the determination of the absorption coefficients of heavy atoms in the soft X-ray region, a domain in which experiments are still difficult to perform; Au (Jaeglé & Missoni, 1966), Bi and Pb (Jaeglé *et al.*, 1967), Pt and Ta (Jaeglé *et al.*, 1968*a*). Combination of experiments and calculations showed that the variation of the absorption coefficients of these atoms are related to photoionization in their 4*f* and 5*d* shells. They always used their bent-crystal spectrometer, which they moved back home only in 1971. They visited Frascati for another reason too: to keep informed on

[3] The first use of synchrotron radiation in Europe. Most technical work at Frascati was carried out by the technician Henri Ostrowiecki, a member of the French team; actually, he was the person who recorded the very first spectra on 6 May 1963 (personal communication).

[4] The description of the instrument and its schematic drawing [*cf.* Fig. 1 of Jaeglé (1966); Fig. 1 of Cauchois *et al.* (1963*a*)] fits well with the improved version of a bent-crystal spectrometer Cauchois had devised in the 1920s and was re-described, in a better modification, by Cauchois (1945).



(*a*) (*b*)

Figure 4
(*a*) Microphotometric recording of two photographic films obtained using the (100) reflection of quartz in the 8 Å region of frequency (ν): the upper one, marked (*a*), was recorded on a 40 µm-thick beryllium plate set in the absorber position of the spectrometer and shows that the synchrotron radiation was too weak to pass through such a thin plate of a light atom; the lower one, marked (*b*), was recorded on a 7 µm-thick Al foil at the same position. In this second image one sees two regions indicated as I and II, separated by the very strong absorption discontinuity (actually a strong change in the film darkening), which corresponds to the Al *K*-edge at 7936 Å in frequency (or 1562 eV in energy). Now the abscissa scale would be the energy (E), and the edge value 1559.6 eV. The $\sim$2.5 eV energy difference is a measure of the accuracy attained by these pioneering measurements (Williams, 2001; the current reference value is 1559.6 eV). The spectra have been recorded during a sequence of exposures during 80 s; they are manifestly overexposed, with the exception of region II. The picture is fuzzy in the original (Cauchois *et al.*, 1963*a*, p. 411, Fig. 2, left) and has not been retouched. (*b*) Microphotometric recording as a function of frequency ν of the blackening of a film with the L_{III} main absorption edge of copper at 13.3 Å (corresponding to 932 eV in energy E, to be compared with the current value of 932.7 eV; Williams, 2001). The precision of the measurement is much better, although no details on the thickness of the foil nor on exposure time are given. The original picture is full of fine spots all over (Cauchois *et al.*, 1963*a*, p. 411, Fig. 3), possibly due to dust present in the developing or in the fixing bath, and has not been retouched. The edge appears as a fairly large dip over a downward-sloping absorption line, as typically happens when converting plate blackening into graphs. The smaller dips nearby may be related to trace impurities of other atoms.

new results obtained on the electron synchrotron by their Italian colleagues. Among these results, two are definitively worth mentioning. A cooperation between ISS and Rome University recorded what is likely to be the first complete spectrum measured in Europe from a synchrotron radiation source, *i.e.* the *K*-edge spectrum of Al metal thin foil up to 70 Å, $\sim$80 eV above the edge [Fig. 3 of Balzarotti *et al.* (1970)]. The researchers did not use the French spectrometer but a home-made set-up consisting of a McPherson monochromator working in the Rowland geometry. The availability of this apparatus probably accelerated the withdrawal of the French spectrometer from Frascati. Secondly, a group from Rome University measured the reflectivity of KCl up to 45 eV above the edge, thus extending the range of previous measurements for light atoms (Balzarotti *et al.*, 1974*a*); as a matter of fact, this work included measuring the absorption coefficient to unprecedented energies that include the 3*p* levels of K^+ and the 3*s* levels of Cl^-. Moreover, the French visitors drew better experience from the many improvements to the electron synchrotron and to detectors being made by the Italian machine group (Balzarotti *et al.*, 1974*b*).

The French–Italian cooperation was successful for an additional reason: it helped Yvette Cauchois to pursue her objective of pressing the French authorities to build a similar synchrotron radiation laboratory in their national, already existing, facility at the Anneau de Collision d'Orsay (ACO). It took her ten years to reach this goal and see the French synchrotron radiation laboratories (LURE) pursue high-quality research independently (Cukier *et al.*, 1974; Jaeglé *et al.*, 1974). These excellent laboratories owe much of their success to the advice of several Italian groups operating at Frascati (Salvini, 1962; Balzarotti *et al.*, 1974*b*). Indeed, the potential research on photons extracted from the electron synchrotron had gained the interest of the Italian community, and attracted researchers from CNR and universities towards XAS studies. Unfortunately, the electron synchrotron was disassembled in 1975 and Italian researchers had to await the availability of the first-generation storage ring ADONE, which started operation in 1978.

Over a short period of time several countries developed their own storage-ring synchrotron radiation facility; in USA [*e.g.* SSRL at Stanford University campus, where the first spectrum taken by synchrotron radiation extracted from a storage ring was made in 1974 (Kincaid & Eisenberger, 1975)] and in European countries [*e.g.* SRS, Daresbury (UK); DORIS, Hamburg and BESSY, Berlin (Germany); DCI, Paris (France); *etc.*], including Italy and France (Bonnelle & Dhez, 2013). Nonetheless, the spirit of international cooperation started by Cauchois and Ageno that fostered the Frascati electron synchrotron never faded away; actually, it triggered a greater cooperation that later would promote the establishment and operation of the ESRF, the European Synchrotron Radiation Facility at Grenoble, today the most brilliant synchrotron radiation laboratory in Europe.

Figure 5
The emission line of aluminium at 8.3 Å (= 1493 eV) split into a doublet by the quartz bent-crystal analyser (Cauchois *et al.*, 1963*b*, p. 1243). The exposure time was 2.5 min, the detector a photographic film, the blackening of which was then converted into a graph by a microphotometer. The current emission-line energies are 1486.70 and 1486.27 eV.

We thank the many colleagues who recalled on our behalf the pioneering days when synchrotron radiation first met X-ray absorption spectroscopy and set forth a fruitful collaboration between Italian and French physicists; particularly, G. Salvini, P. Salvadori, M. Grandolfo, A. Bianconi, E. Burattini, R. Habel, G. Battimelli, P. Dhez and, last but not least, G. Missoni. They all witnessed an almost forgotten yet wonderful period of scientific development, and were so kind as to share their reminiscences with us. We also thank A. Michalowicz for additional information. The critical advice of S. S. Hasnain, P. Pianetta, and J. F. van der Veen helped us greatly, as also did the accurate reviews of two referees. The text has been linguistically improved by Ms J. P. Moliterno.

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