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

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Abstracts

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 by the French-Italian group made of Y. Cauchois, C. Bonnelle and G. Missoni in April 1963.

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1. The Italian synchrotron facility of Frascati

Who first envisaged and first detected synchrotron radiation is a matter of controversy among Science historians. According to some of them, the first theoretical demonstration that electrons orbiting within a magnetic field dissipate photons arose in the minds of Russian physicists D.D. Ivanenko and I.Ya. Pomeranchuk in 1944; according to others, the first scientist who put it on paper was V.I. Veksler, another Russian and in the same year. Similarly, E.M. McMillan is credited to have built the first electron synchrotron in 1945 at Berkeley, CA, and to have given the name to this new type of particle accelerator; yet, the first observation of a bright arc of light (i.e., of visible synchrotron radiation) was made at Schenectady, N.Y., 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 [1]. There is also a little controversy about the first synchrotron operating in Europe: perhaps there was one somewhere in Moscow, USSR, but the information on it is practically none because it fell among cold-war secrets; by contrast, it is certain that the first electron synchrotron in operation in Western Europe was that at Frascati near Rome, Italy [2, p. 15].

1.1. Establishing the synchrotron

The proposal to build an accelerator of 500-1000 MeV power in Italy had come from Gilberto Bernardini (January 19, 1953); the first funding came from a share of the yearly grant the "Istituto Nazionale di Fisica Nucleare" (INFN), at that time a division of "Consiglio Nazionale delle Ricerche" (CNR), received from the Italian government. 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 a much larger support from the state. The directorship of the project was conferred from the very beginning to Giorgio Salvini, who took upon himself the responsibility of building an electron synchrotron instead of a linear accelerator. Frascati was chosen to host it. It took four years to prepare the laboratory hall at Frascati, while the design of the apparatus started in summer 1953 at the Institute of Physics of Pisa University. Later, in May 1955, the "Sezione Acceleratore" moved from Pisa University to Rome University to follow the on-going works. The final step occurred in summer 1957, when a truck travelled ten times from Pisa to Frascati to transport the components already built. This is the year when the "Laboratori Nazionali del Sincrotrone" (LNF) of "Istituto Nazionale di Fisica Nucleare" (INFN) were 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 February 9, 1959, the foreseen further target being to reach 1100-1200 MeV [3].

Congruently with the INFN *raison d'être*, the electron synchrotron (Fig. 1) was primarily dedicated to research in nuclear physics, such as the pion-nucleon resonance, the electron-pion diffusion, the neutral pion decay and the properties of meson η. Indeed, the first users had nothing else in mind but nuclear physics, and concentrated all their experimental skill on

researches related to it. Their results were interesting and widely appreciated, although they had to avoid some key experiments not to stress the machine, since the attitude of the politicians towards research had soared in the meantime (1963-67) and money was running short.

During operation, an electron synchrotron generates an incoherent emission of photons that are due to the slowing down of the circulating electrons, and these photons in turn assume the shape of a quasi-monochromatic, homogeneous and ca. 70% polarized beam of high resolution ($> 10^{-3}$) when interacting with a single crystal of diamond [4]. The availability of such 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 energy [5], should have offered the Italian physicists the possibility of entering a field of research they had never approached before i.e., X-ray spectroscopy. They did not, and this turned out to a profit for Science, because it generated a first, significant international collaboration in Europe.

1.2. Yvette Cauchois and her X-ray absorption spectroscopy interests

Yvette Cauchois (1908-1999) was one of the very few women of her time who graduated in physics (1928) and could enter the Sorbonne University of Paris (France) to earn "troisième cycle" (1933) under Jean Perrin, the 1926 Nobel Prize in Physics. She dedicated to Physics all her life. Actually, she engaged herself in X-ray spectroscopy: a rather restricted field of physical research, which, however, had had recent, illustrious tradition in France and is now identified as X-Ray Absorption Spectroscopy (XAS). Indeed, Maurice de Broglie [6], concurrently with Julius Herweg in Germany [7], had been the first to detect and consider the series of dark and light bands, which follow the X-ray absorption edge of an element impinged by X-rays; he named spectrum these bands, and XAS the branch of Science studying the properties of such a spectrum. His commitment to 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. On 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 outstanding diffraction pictures that turned out from natural and synthetic crystals, when directly impinged by the characteristic X-ray radiation beam emitted by a discharge tube. Consequently, in France, Maurice de Broglie was almost alone in his effort of forwarding XAS during the years '10 and '20, when most if not all "Big Science" was being carried out by XRD (e.g., M. von Laue, W.L. Bragg, P. Debye, P.P. Ewald, etc.). Actually XAS was studied, but only as a second-rank field and mostly at Lund (e.g., M. Siegbahn) and Harvard universities (e.g., D. Coster, R. Kronig), with the scattered theoretical support of some other scientist (e.g., W. Kossel).

In France, nobody opposed Yvette Cauchois' decision to dedicate herself almost entirely to XAS. She was left free to work, but she was mostly alone with little support. During the years

‘30s 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 (Fig. 2), she took up writing books for the education of new physicists and published her experimental results, her most remarkable contribution being a book in the form of tables [8] that rapidly became an internationally recognized reference. Moreover - as a rather rare case among the full professors at Sorbonne University in Paris - she did not give up personal research entirely, and induced many valuable students and researchers to go on with X-ray research together with her, always pushing them to look for new, innovative aspects of an apparently endless open field. Enough for Cauchois’ merits and achievements: they are better described elsewhere [9]. Let’s go back to the very subject of this paper: the fiftieth anniversary of the first synchrotron radiation derived X-ray absorption spectrum.

1.3. Beginning the French-Italian cooperation on synchrotron

In 1961 Yvette Cauchois had had one of her brilliant ideas. As the leader of the Laboratory of Physical Chemistry of Paris Faculty of Science, she started a co-operation with Mario Ageno (Fig. 3), at that time the leader of the Physical Laboratory of “Istituto Superiore di Sanità” (ISS) in Rome. The initiative was probably triggered by Ugo Fano, at that time at the U.S. National Bureau of Standard in Washington DC, where the 180 MeV SURF electron synchrotron was in operation producing the first significant absorption spectra in the VUV range. Fano was visiting Frascati on summer every year and was a staunch supporter of the local new facility. He and his co-worker J.W. Cooper visited Cauchois’ laboratory in Paris in 1962 or 1963 and brought exciting information on the potential of the new machine [9]. The Italian-French agreed joint program would mainly concern the determination of the photoionization cross sections of elements with high atomic number, which, at that time, was possible only using 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. [3]); therefore, they had access to the synchrotron, although with some limitations for time because, at the suggestion of E. Fermi, the final injector used had been the more powerful commercial Van der Graaf one. After more than one year’s delay used to set up in Paris a grating spectrograph [10] 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 moved to Frascati with her apparatus and with a group of students, researchers and technicians, set everything up next to the ring and started making good use of the limited beam time allotted to her. Her first paper [11], written in cooperation with her pupil Christiane Bonnelle and with Guido Missoni, an ISS researcher whom Ageno had committed to the job, describes carefully the entire set up and gives information on the first results, but it tells us little about the difficulties encountered because of the lack of a beam line built to properly collect the radiation made available by the accelerator

and of the instability of the source itself. Such difficulties are described vividly by Pierre Jaeglé, Cauchois' main co-worker, in a recollection written 25 years afterwards: the available space was "*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*" [12, p. 22].

2. Experimental results

2.1. Absorption spectra. The first scientific information gathered by Cauchois and co-workers in their pioneering studies at Frascati electron synchrotron was twofold: a) the radiation emitted at 1 GeV covered the electromagnetic spectrum from X-rays to visible, with maximum intensity at about 10 Å (i.e., ca. 1240 eV), thus "*interéssante*" [11, p. 409] when compared with the bremsstrahlung emitted by conventional sources, which at this energy may start fading away; it was fit particularly for research on Biology [10, p. 409]; b) the use of the bent-crystal spectrograph coupled with photographic recording was possible despite the small space allotted [11, p. 409] and the large instability of the synchrotron beam. The instrument Cauchois had installed at Frascati [10-12] operated under vacuum in the 2 to 25 Å range (ca. 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 really exciting [11, p. 412); not so much, possibly, for the accuracy of the measured absorption edges (Al-K = 1566 eV and Cu-L_{III} = 930 eV [13], with the former showing up only as a discontinuity in the overexposed plate (Fig. 4a), but the second one being a clear jump followed by a fine structure (Fig. 4b). What it appeared to be most interesting was the significantly shorter exposure time, which turned out to be of the order of "*quelques dizaines*" [11, p. 402] up to 50 times [15, p. 1243] shorter than what could be expected from the best conventional discharge tube.

Moreover, Cauchois et al. [11, 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 they were aware of (the Be-K edge at 112 eV and the Al-L_{III} edge at 72.8 eV [16], and the absorptions of gases in the energy range 26 to 69 eV [17]), both obtained in the USA, were at much lower energy. Surprisingly, Cauchois forgot, or was unaware of the very early experiment by which Johnston and Tomboulian [18] in 1954 had given to the world physics community the first demonstration of a XAS spectrum recorded from polychromatic synchrotron radiation. Moreover she did not refer to the theoretical paper where Parrat [19] had mathematically shown how much gain XAS would get from using synchrotron radiation instead of conventional X-ray sources.

2.2. Emission results. To finish up the story, a short addition is appropriate, although it is out of the main theme. In their second paper, Cauchois et al. [15 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 minutes time and by using a gypsum crystal they could register the entire $K\alpha$ emission line at 1490 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 development

The French scientists showed quite openly that they were happy with their results, as they kept visiting Frascati over the years and for several times [20-23]. Their experiments, mostly performed together with researchers of Ageno's group, were focused on the determination of the absorption coefficients of heavy atoms in the soft X-ray region, a domain in which it is still difficult to perform experiments: Au [20], Bi and Pb [21], Pt and Ta [22]. By combining experiments and calculations they could demonstrate that the variation of the absorption coefficients of these atoms are related to photoionization in their 4*f* and 5*d* shells. They moved back their spectrometer only in 1971. In the meantime they had visited Frascati for another reason too: to keep informed on new results obtained on the electron synchrotron by their Italian colleagues. Among these, two are definitively worth sorting out. 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 = ca. 80 eV above edge [5 Fig. 3]). They did not use the French spectrometer, but an optical system designed to match a McPherson monochromator working on the Rowland circle geometry. Later, 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 [24]; as a matter of fact, this work included measuring the absorption coefficient to unprecedented angles that include the 3*p* levels of K^+ and the 3*s* levels of Cl^-. Moreover, the Frenchmen drew better experience from the many improvements to the electron synchrotron and to all recording apparatus of it being made by the Italian machine group [25].

The French-Italian cooperation was successful for one additional reason. For all the fairly long time span it lasted, Yvette Cauchois kept pursuing her major aim, which was at pressing the French authorities to build a similar synchrotron radiation laboratory in their national, already existing facility "Anneau de Collision d'Orsay" (ACO). It took ten years to her to reach satisfaction and to see the French synchrotron radiation laboratories (L.U.R.E.) set there to pursue high-quality research by their own means [26, 27]. The excellent outcome of these laboratories owns much to the advice of the several Italian groups, which had meanwhile developed at Frascati (cf. [3] and [25]). Indeed, the potential research to be carried out by photons extracted from the electron synchrotron had entered the interest of the physical

community, and the Frascati laboratories attracted people from CNR and universities towards synchrotron radiation and, in particular, to XAS studies. Almost simultaneously, because of the approval at Frascati of a storage ring, the physics community started looking for other, more intense and more reliable X-ray sources. The Italian researchers repeatedly went to L.U.R.E. and tested their prototype storage ring (AdA) there, so that both Frenchmen and Italians kept moving back and forth even after the electron synchrotron machine was disassembled (1975) and replaced by a first-generation large storage ring (ADONE), which entered operation for high-energy physics researches in 1978.

Over a short period of time several storage rings entered into operation in many countries [2], both in U.S.A. (e.g., SSRL, Menlo Park, CA, where the first spectrum taken by synchrotron radiation extracted from a storage ring was collected contemporary to Italian researchers in 1974 [28], and in several countries of Europe (e.g., SRS, Daresbury, U.K. [29]; DORIS, Hamburg, Germany [30]; etc.). Italy and France also developed their own synchrotron radiation facilities, and yet the spirit of mutual cooperation started by Cauchois and Ageno through the benefit of Frascati electron synchrotron never faded away [31]. Actually, it triggered and helped creating an enormously greater cooperation that later have been used to built and operate ESRF: the European Synchrotron Radiation Facility at Grenoble, the most powerful synchrotron radiation laboratory in Europe.

Acknowledgements. 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. Among them we point out 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 and yet wonderful period of scientific development, and were so kind as to share their reminiscences with us. The critical advices of S. Hasnain, P. Pianetta, and J.F. van der Veen helped us greatly. The Ageno's picture (Fig. 3) was kindly made available by "Archivio storico fotografico" of "Settore Attività Editoriali dell'Istituto Superiore di Sanità, Roma", which also allowed its publication.

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experiments of physics 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-ray and the production of the monochromatic beam.
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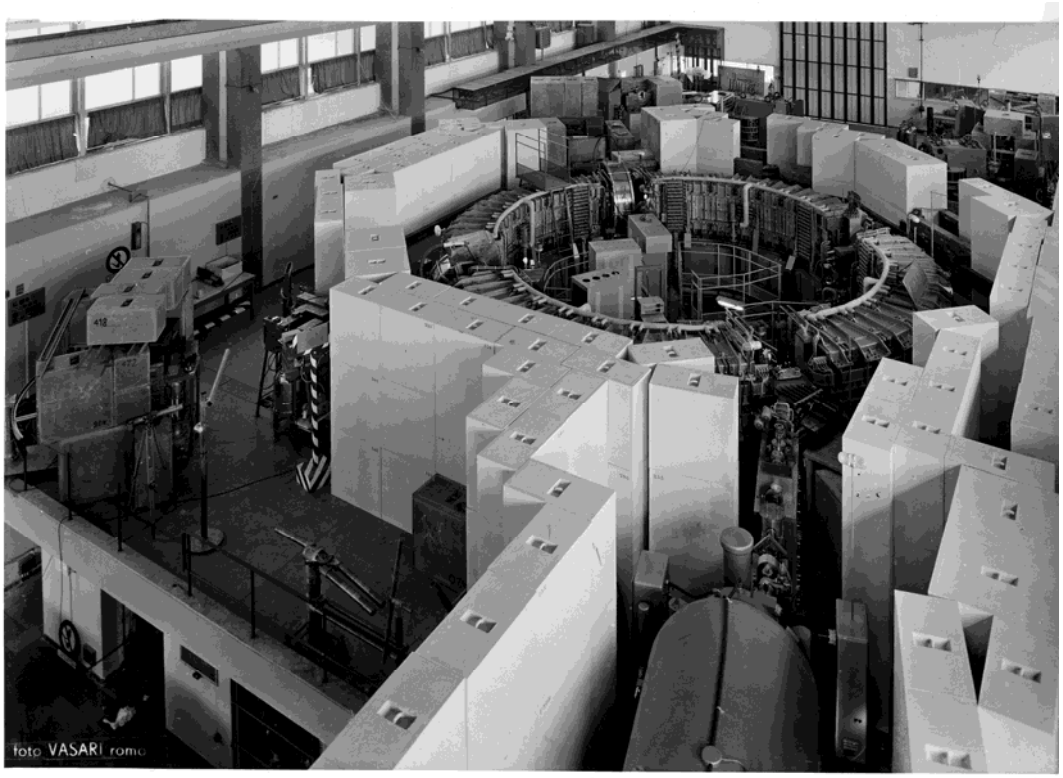

Fig. 1. View of the Frascati electron synchrotron, in operation from 1959 to 1975, with all the beam lines derived from it. The Italian-French cooperative experimental set up was located in the left-hand side sector.

Fig. 2 Yvette Cauchois (1908-1999)

Fig. 3 Mario Ageno (1915-1992).

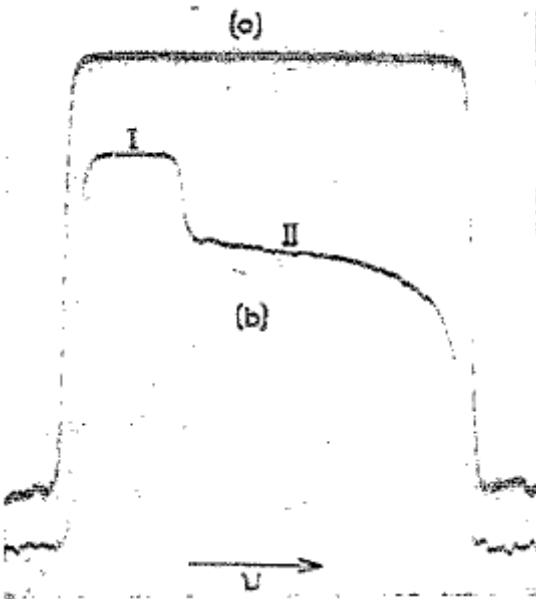

Fig. 4a. Micro photometric recording of the reflection (100) of quartz in the 8 Å region, obtained with a 40 μm-thick beryllium absorber (above) and a 7 μm-thick Al absorber (below). One sees the very strong discontinuity at 7935 uX (= 7919 Å = 1490 eV) that corresponds to the Al *K*-edge.

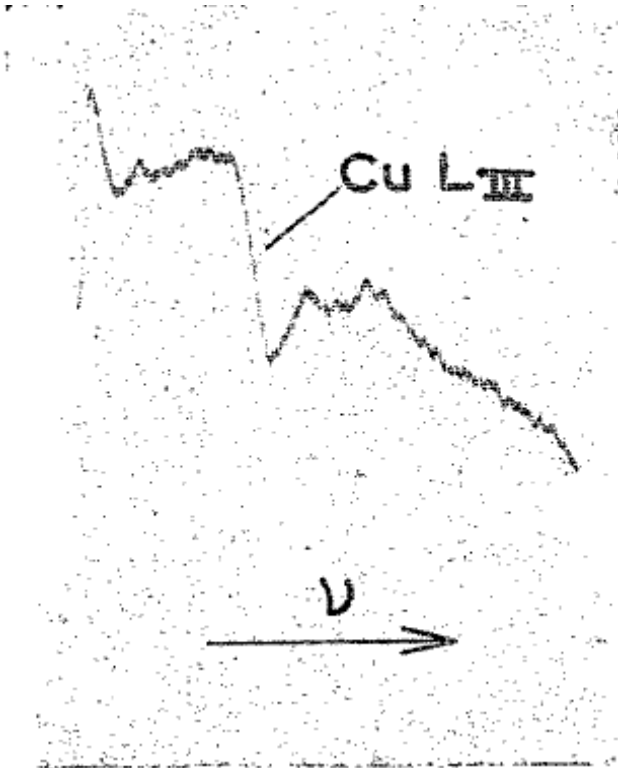

Fig. 4b. Micro photometric recording of the L_{III} spectrum of copper showing the region of the main absorption edge at 13.3 Å (= 930 eV).

Fig. 5. The emission line of aluminium at 8.3 Å (= 1490 eV), the line being split into a doublet by the resolution intrinsic in the quartz bent-crystal analyser.