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Historical Article

Estonian Scientist in USSR (Memories and Reflections about Endel Lippmaa, 1930-2015)

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Abstract. Endel Lippmaa - Estonian physicist and chemist was one of the pioneers in the development and application of NMR method (1950s-1970s). He had to work in conditions when Estonia was part of the USSR as the Estonian SSR and, nevertheless, the development of science in the republic was not interrupted. He later became an academician and vice-president of the Academy of Sciences of the Estonian SSR. The important role of Lippmaa in the development of nuclear magnetic resonance and other areas of chemical physics in Estonia, the USSR, and around the world is considered. The article also contains the reminiscences considering personal contacts with Lippmaa of the famous Russian chemist Yu.A. Ustynyuk. In 1994 Lippmaa was awarded the AMPERE Prize for original research in high-resolution NMR of solid-state. Perhaps in more favorable conditions, in contact with other eminent scientists who worked abroad, he would have achieved even greater results, but such contacts were during a long time difficult or impossible for him.

Keywords: Endel Lippmaa, Estonia, Soviet period, proton magnetic resonance, carbon-13 NMR, NMR high resolution in solid, ion cyclotron mass spectrometry.



Figure 1. Endel Lippmaa (Photo 1970-s. Author archive).

HISTORICAL BACKGROUND (BASED ON MATERIALS AVAILABLE ON THE INTERNET)

Scientific and educational institutions in the history of Estonia

Estonia became a battleground for centuries where Denmark, Germany, Russia, Sweden and Poland fought their many wars over controlling the important geographical position of the country as a gateway between East and West.

The «Russian era» from the 1720s to the First World War, it was a time when German elites prevailed and even had some autonomy in the Baltic countries.

However, in those same years, the formation and consolidation of the Estonian nation, its culture and language took place. There were also tendencies in Russification of Estonia. The impact of this was that many of the Baltic German legal institutions were either abolished or had to do their work in Russian – a good example of this is the University of Tartu. It was founded under the name of *Academia Gustaviana* in 1632, shortly before the king's Gustavus Adolphus death on 6 November in the Battle of Lützen (1632), during the Thirty Years' War (1618–1648). The University of Tartu moved to Tallinn in 1656, and in 1665, it closed down. The university was reopened by the Baltic Germans in Estonia in April 1802. The language of instruction at Dorpat (Russian – “*DERPT*”) was German from 1802 to 1893. During that time, Dorpat had a dual nature in that it belonged both to the set of German-language and Russian universities. Tartu was a multicultural crossroads with strong representation of Russians, Germans and Estonians. Orthodox, Lutherans and Jews, scientists and humanists, all were quite active at the city's university. Since Estonia became independent in 1918, the University of Tartu has been an Estonian-language institution since 1919. The university was named Ostland-Universität in Dorpat during the German occupation of Estonia in 1941–1944 and Tartu State University (Estonian: *Tartu Riiklik Ülikool*) in 1940–1941 and 1944–1989, during the Soviet rule, although Estonian remained the principal language of instruction, some courses were taught in Russian, with several Russian curricula.

Before Estonia independent young people from Estonia had received their specialist education in St. Petersburg, Germany or Riga. Opportunities had to be sought for engineering-minded people to acquire an Estonian-based education which was adapted to local conditions and needs; Estonia was in the process of establishing itself as an independent country.

On 17 September 1918, the Estonian Engineering Society opened an Estonian-based engineering school

named Special Engineering Courses. That date has been recognised as the founding date of Tallinn University of Technology. Programmes were offered in mechanical, electrical, civil and hydraulic engineering, shipbuilding and architecture. In 1919, the school became the private Tallinn College of Engineering, which in 1920 was declared a state institution. Teachers' efforts to develop an Estonian terminology for science and technology proved fruitful and the first engineering books were published. In 1923, the first engineering graduation theses were defended in Estonia. In the same year, a state laboratory of materials testing opened for research work.

By the 15 September 1936 Act of the Head of State, the school was granted university status, and named Tallinn Technical Institute. The institute had two faculties: civil and mechanical engineering and chemistry and mining. In 1938, the name Tallinn Technical University (Tallinna Tehnikaülikool, TTÜ in Estonian) was effective. In 1940 the Faculty of Economics, in 1958 the Faculty of Power Engineering and in 1965 the Faculty of Control Engineering were founded.

The Estonian Academy of Sciences was established in 1938. When Soviet troops entered Estonia the Academy was dissolved on July 17, 1940. In June 1945 it was reestablished as the *Academy of Sciences of the Estonian SSR* (Estonian: *Eesti NSV Teaduste Akadeemia*). In Soviet times, it consisted of a central library and four divisions containing 15 research institutes as well as other scientific societies and museums. In April 1989, shortly before Estonian independence, the academy regained its original name of *Estonian Academy of Sciences*. The Estonian scientific community was least affected by the repressions carried out by the Soviet authorities and directed against country's leading politicians, military officers, and many small private owners.

Remarks on the history of Estonia in the Soviet period

After Stalin's death in 1955 the TV Centre was built in Tallinn; it began TV broadcasts on 29 June of that year. One positive aspect of the post-Stalin era in Estonia was the regranting of permission in the late 1950s for citizens to make contact with foreign countries. Ties were reactivated with Finland, and in the 1960s, a ferry connection was opened from Tallinn to Helsinki and Estonians began watching Finnish television. This electronic “window on the West” afforded Estonians more information on current affairs and more access to Western culture and thought than any other group in the Soviet Union. This heightened media environment was important in preparing Estonians for their vanguard role in extending perestroika. See for example [1].

The Tallinn Song Festival Grounds, the venue for the song festivals, were built in 1960. Communist Party membership vastly expanded its social base to include more ethnic Estonians. By the mid-1960s, the percentage of ethnic Estonian membership stabilized near 50%. On the eve of perestroika the ECP claimed about 100,000 members; less than half were ethnic Estonians and they totalled less than 7% of the country's population.

Only after the Khrushchev Thaw period of 1956 did healthcare networks start to stabilise. Due to natural development, science and technology advanced and popular welfare increased. All demographic indicators improved; birth rates increased, mortality decreased. Healthcare became freely available to everybody during the Soviet era.

From the history of the scientific dynasty of Lippmaa

The ancestors of Endel Lippmaa during the reign of the German elites in Baltic (during the eleventh and beginning of the twentieth centuries) bore the name "Lippmann". An outstanding scientist, botanist and chromatographer born at Riga in 1904, Endel's father Theodor Lippmaa was registered under this name, but changed this name to consonant Estonian after moving to the territory of independent Estonia. Unfortunately, the father of Endel Lippmaa tragically died on January 27, 1943 in Tartu as a result of the bombing of the city by Soviet aircraft.

Endel Lippmaa left two sons Jaak and Mickey, who continued the scientific dynasty.

INTRODUCTION

In the very beginning of the 1960-s among Soviet physicists and chemists formulated a task to equip chemists with new instruments suitable for studying nuclear magnetic resonance spectra (NMR). European and American colleagues have already solved this problem (at the technical level of that time) by the efforts of the American company Varian, with which some fairly weak European firms and Japanese JEOL competed. Already in those years on the so-called «world level», it was impossible to imagine the identification of a new chemical compound without NMR spectra (most often ^1H , sometimes ^{19}F , ^{31}P , ^{11}B , etc.). However, in the USSR, NMR spectrometers were exotic, often useless for chemists due to their technical characteristics. They were manufactured and debugged in separate laboratories with rather outdated electromechanical workshops. The Estonian engineer and scientist Endel Lippmaa who

graduated in 1953 from the Tallinn Polytechnic Institute and worked there, decided to devote his scientific activity to NMR. But as early as 1956, he became a candidate of technical sciences, having defended a thesis in the field of chromatography (The undoubted influence of the works of his father T. Lippmaa). Endel Lippmaa's activities for a considerable time were closely connected with the functioning of the entire Soviet scientific community, especially the part of it that was engaged in the technique and application of magnetic resonance. Subsequently, Lippmaa contributed to the development of ion-cyclotron resonance mass spectrometry (ICR MS)

FIRST NMR SPECTROMETER IN ESTONIA. THE BEGINNING

After five years of painstaking work in 1962, E. Lippmaa joined a cohort of NMR radiospectroscopists. He published a series of works on the construction of a high-resolution PMR spectrometer (proton magnetic resonance spectrometer) in the Works of the Tallinn Polytechnic Institute (TPI) [2,5]. The table shown in Fig. 2 was apparently photocopied from the work of Lippmaa [3], which was then kept in the «Literature in the Languages of the Peoples of the USSR» hall of the Russian State Library preserved from Soviet times.

Although the article was published in Russian (annotations in English and Estonian), Estonian journals were supposed to be in the above named hall. The notes were on the text of the copy we received, supplemented by the author of the cited article by hand. For the first time this photocopy with notes was published in our book [6, p. 579]. We clarified the notes and allow ourselves to bring a table with the old and our necessary new additions.

Таблица 1

Разрешение некоторых ЯМР-спектрометров с магнитами собственной конструкции¹

№	Спектрометр	Разрешение по $-\text{OH}^*$	Разрешение по $-\text{CH}_2^{**}$
1956 1	Арнольд [4] РИГС. РЕН. 102/56	$1,3 \cdot 10^{-8}$	$1,3 \cdot 10^{-8***}$
1957 2	Примас [10] ИРА 30, 305	$2,5 \cdot 10^{-8}$	$2,1 \cdot 10^{-8****}$
1964 3	Владимирский [14] ПТФ, 6, 4, 59	—	$9,7 \cdot 10^{-8****}$
1965 4	Быстров, Декабрун и др. [8]	$1,3 \cdot 10^{-7}$	$1,7 \cdot 10^{-7}$
1965 5	Самитов [7]	$1,8 \cdot 10^{-7}$	$2,3 \cdot 10^{-7}$
1965 6	ЯМР-спектрометр со спингенератором [10, 20]	$1,5 \cdot 10^{-7}$	$1,7 \cdot 10^{-7}$

* Относительная ширина линии гидроксильной группы подкисленного этилового спирта на полувысоте.
 ** Относительная ширина центральной линии триплета метильной группы подкисленного этилового спирта на полувысоте.
 *** Одна линия дублета. Ширини линий измерены по опубликованным спектрам.
 **** Без вращения образца.

Figure 2. A photocopy of the table from the Lippmaa article that compares the characteristics of the instrument manufactured in his laboratory and the characteristics of the instruments of his predecessors.

Table 1. Resolution of some NMR spectrometers of own design.

No	Year of edition	Constructors	Reference	Resolution by OH *	Resolution by - CH ₃
1	1956	Arnold	<i>Ph.R. 10.2.136</i>	1.3·10 ⁻⁸	1.3·10 ⁻⁸ ***
2	1957	Primas	<i>H.P.A. 30.515</i>	2.5·10 ⁻⁸	2.1·10 ⁻⁸ ***
3	1961	Vladimirsky	<i>P.T.E 6.C.459.</i>	–	9.7·10 ⁻⁸ ****
4	1961	Bystrov, Dekabrun et al.	<i>See P.T.E 1961. N1. C. 122</i>	1.3·10 ⁻⁷	1.7·10 ⁻⁷
5	1961	Samitov	<i>See P.T.E. 1961. № 5. C. 100</i>	1.8·10 ⁻⁷	2.3·10 ⁻⁷
6	1962	<i>Lippmaa et al.</i>	<i>[2, 4]</i>	1.5·10 ⁻⁷	1.7·10 ⁻⁷

Additions are in italics. * The relative line width of the hydroxyl group of acidified ethyl alcohol at half-height; ** The relative width of the center line of the triplet of the methyl group of acidified ethyl alcohol at half-height; *** One line of doublet. The line widths are measured from published spectra; **** Without sample rotation usually applied in NMR.

Abbreviations: *Ph.R.* – *Physical Review*; *H.P.A.* – *Helvetica Physica Acta*; *P.T.E.* – *Russian journal "Pribory i Tekhnika Eksperimentalnaya" ("Instruments and Experimental Technique")*.

From the experience of cooperation with a specialist in the development of semi-industrial NMR spectrometers A.N. Lyubimov and acquaintance with his attempts to “embed” his development at the plant [7] we know that the fatal limit of 1.5·10⁻⁷ is easily overcome with adequate heat treatment of the pole pieces of a magnet (annealing in a *hydrogen atmosphere* as an example), which is inaccessible, as a rule, for laboratory developers.

We clarified the notes [6] and allow ourselves to bring a table with the old and our necessary new additions (see Table 1).

Among the staff of Lippmaa, as a true leader, you can identify assistants (A.Sügis, Abira Olivsson), associate (V. Sinevee) and followers (T. Pehk, M. Mägi). Lippmaa went his own way, he studied all the available data from the experience of his predecessors, immediately chose an electromagnet, and not a permanent magnet. He himself checked the magnetic susceptibility of materials for manufacturing the details of the NMR sensor [4], applied the method of stabilization of resonance conditions along one of the lines in the NMR spectrum. Moreover, as it became clear later, for this team the creation of a high-resolution NMR spectrometer for protons was only a necessary intermediate step.

At the same time, Lippmaa was not going to create a model of the NMR spectrometer that Soviet industry could produce. His aim at the first stage was to create his own laboratory of chemical NMR radiospectroscopy.

Sector of Physics at the Institute of Cybernetics

In the early 1960's. (at the end of 1961), Lippmaa and his team became employees of the new academic

Institute of Cybernetics (Academy of Sciences of Estonia). Institutes with such a name were created in several Academies of Sciences of the Union Republics of the USSR, for example, also in Georgia, but this does not mean that they were engaged there only cybernetics.

Creation of a new Institute affected availability of new equipment. In particular, the Lippmaa's laboratory in a fantastic way received a Japanese (JEOL) magnet providing the resolution necessary for proton resonance, and a Finnish storage device (the simplest computer). The research sector of new Institute, which was headed by Endel Teodorovich, had the name “Sector of Physics”. And, because already at the first stage of work at this institute in the laboratory (Sector) Lippmaa began to master the application to the programming of experiment and accumulation of experimental data on digital computing devices, this could justify the appearance of such a subdivision in the Institute of Cybernetics. The Institute received a new building in the very center of Tallinn, comfortable and decorated in a slightly abstract style, reminiscent of the design style of some of Moscow and Novosibirsk institutes. The Institute was headed by a relatively young (specialist in the field of mechanics, born in 1918, academician of the Academy of Sciences of the Estonian SSR) Niil Alimäe. The support of the undoubtedly talented and promising Endel Lippmaa, who graduated from the Estonian Higher Educational Institution (TPI) after the war and there defended his thesis, points to the continuity of the development of science in Estonia for three periods: pre-Soviet, Soviet and post-Soviet. In the new Institute, the Lippmaa laboratory intensively mastered the methods of the double proton-proton NMR and took the first steps in the development

of the rare isotopes ^{13}C and ^{15}N NMR. A series of publications in these fields appeared in 1965 - 1969 in the Proceedings of the Estonian Academy of Sciences [8–23]. Some of these publications were in English and one of them (together with V. Sinivee) is in Estonian [14], which demonstrated the availability of scientific terminology in the new field of knowledge in this language. For example, «*tuuma magnetilises toplet-resonantsis*» is «nuclear magnetic double resonance»..

One of the founders of carbon ^{13}C NMR spectroscopy

Since 1965, the most authoritative specialists in organic chemistry and the heads of the General and Technical Chemistry Division of the Academy of Sciences of the USSR began to cooperate with Lippmaa for a simple reason: the tools of the Lippmaa's laboratory in the 1960s made it possible to carry out experiments «unattainable even for remarkable imported instruments, which were acquired for the Moscow institutes» (E. Lippmaa, speech at the First All-Union Symposium on NMR, Tallinn, 1967). Endel Lippmaa in the 1960s received an opportunity to contact chemists from the German Democratic Republic, Finland and Sweden, he established strong ties with Soviet chemists. His laboratory was one of the three in the world that laid the foundation for ^{13}C carbon spectroscopy. He became the third creator (after Lauterbur [24] and Holm [25]) of the experimental base for NMR spectroscopy of a rare carbon isotope. In 1970, three fundamental studies of the carbon-13 NMR of several classes of organic compounds were published by Lippmaa in an international scientific journal jointly with chemists of the various countries [26–28] (and earlier works [13, 16, 23]). In this, he somewhat overtook even the firm of the Varian brothers, leading the implementation of NMR in chemistry.

Outstanding expert in the NMR area

The authority of Lippmaa was extremely high both in the circles of Soviet physicists (the Council of Radio Spectroscopy of the USSR Academy of Sciences, the symposium on NMR and NQR in 1967, the administration of All-Union Schools on magnetic resonance) and Soviet chemists (reports at the general meeting of the Department of General and Technical Chemistry in 1967 and in the Presidium of the Academy of Sciences of the USSR in 1972, cooperation with academicians and their closest associates in the study of chemical structures, regular consultations with colleagues from the chemical faculty of Lomonosov Moscow State University). In 1967, at a

general meeting of the General and Technical Chemistry Division of the USSR Academy of Sciences, Lippmaa demonstrated the capabilities of double proton NMR resonance and NMR of carbon, and nitrogen isotopes to study the structure of chemical compounds. In the same year, Lippmaa met Soviet NMR specialists at the Joint Symposium on Nuclear Magnetic Resonance in Tallinn, and they had the opportunity to get acquainted with the original equipment created in his laboratory. In 1969 Endel Lippmaa defended his thesis for the degree of Doctor of Physical and Mathematical Sciences, specializing in «Chemical physics» in the Alma mater of Soviet chemical physics – the Institute of Chemical Physics of the USSR Academy of Sciences, the topic – The Overhauser Nuclear Effect and the Structure of Organic Compounds. Reviewers (official opponents) were well-known theorist G.V. Skrotzky, creator of one of the first PMR spectrometer in the USSR L.L. Dekabrun, and highly reputable Doctor of Chemical Sciences, Yu.N. Sheinker. A review from a scientific organization was sent by S.A. Altshuler's department from Kazan University.

Lippmaa becomes corresponding member (1972), and academician (1975) of the Academy of Sciences of the Estonian SSR, specializing in «Chemical Physics». In 1980, Endel Lippmaa founded the Institute of Chemical and Biological Physics (now the Institute of Chemical Physics and Biophysics of the Estonian Academy of Sciences). In 1977-1982 he was The Academician-Secretary of the Department of Physical, Mathematical and Technical Sciences of the Academy of Sciences of the Estonian SSR. He was a recognized expert and official reviewer of several dissertations of Soviet specialists in the field of magnetic resonance.

Lippmaa always had clear goals, strategic (to overcome everything and become an expert in a certain field – to start – in the field of NMR application in chemistry) and at each stage – a new tactical goal (create a spectrometer for PMR; master the methods of double resonance, make an essential contribution to the NMR spectroscopy of rare isotopes, master method of high-resolution NMR in a solids). However, more ambitious plans were not alien to him. The first deviation from the “general line” (chemical and analytical NMR), were two papers [29, 30] devoted to a very interesting effect - the polarization of nuclear spins ^{13}C in chemical reaction products in liquids with the intermediate formation of a pair of free radicals (chemically induced dynamic polarization of nuclei, CPN or CINDP). In 1972, the International Conference on CINDP even took place in Tallinn. Endel Lippmaa had however left this direction.

The interests of Lippmaa always went beyond the practice and theory of purely applied NMR and cov-

ered a wide range of problems of chemical and biological physics, as well as physics and chemistry in general. In 1984, on an ion-cyclotron resonance equipment with a field strength of 4.7 T Lippmaa and his colleagues performed an experiment to measure the mass difference between tritium and helium-3 ions. See about it below in a special section.

The scientific authority of Lippmaa in international scientific circles was recognized in 1969 by his inclusion in the editorial board of the newly created journal *Organic Magnetic Resonance*. Subsequently, he led such events as the International Congress on Molecular Spectroscopy (1973), the Congress AMPERE (1978), the VIII All-Union School on Magnetic Resonance (1983).

In 1978 the first in the USSR Congress AMPERE was held in Tallinn. Lippmaa was the head of the local organizing committees for the above-mentioned events, and on behalf of all Soviet science he welcomed foreign guests and apologized that he could not welcome guests as fluently in French, as he welcomed them in Estonian, Russian, German and English.

High resolution NMR of solid state

In the 1970–1990's Lippmaa with his disciples (M.Alla, E.Kundla, A.Samoson, T.Saluvete and others) became classics in the field of high resolution NMR in solids [31–33]. They developed methods for observing NMR of silicon-29, aluminum-27 and others nuclides in powders and polymers. The most important work in this direction was published in 1977 in *JETP Letters* at 1977 [31] (see photocopy of abstracts of English translation of Soviet *JETP Letters* on fig.3). In his report at a joint session of the Branches of the Academy of Sciences of the USSR and the Academy of Sciences of the ESSR [34], Lippmaa reported on the first results of research in this area.

Experiment [31] was rather complicated (actually was a prototype of a distinct variant of two-dimensional spectroscopy) and included both sample rotation (at an angle of $\arccos(1/\sqrt{3})$ to the direction of a polarizing magnetic field), which eliminated the dipole interactions broadening the spectral line of NMR, and strictly periodic (during each revolution) switching on the radio frequency resonant pulses, inverted ^{13}C magnetic moments (Hartman-Hahn sequence type series of resonant π -pulses [35]). The signals obtained after the usual Fourier transform in this case had the form of damped oscillations and again underwent the Fourier transform. The oscillation period was clearly determined by inner anisotropic interactions that were not fully eliminated by rotating the sample (so-called anisotropic part of chemical shift).

Selective determination of anisotropic magnetic interactions from high-resolution NMR spectra of powdered samples

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(Submitted 26 December 1977)

Pis'ma Zh. Eksp. Teor. Fiz. 27, No. 4, 208–211 (20 February 1978)

It is shown that in the case of rapid synchronous rotation of a system in the spin and spatial coordinates it is possible to reconstruct selectively the influence of the anisotropic interactions on the high-resolution NMR spectra of rare nuclei in solids.

PACS numbers: 76.60.Cq, 75.30.Gw

Figure 3. A photocopy of the abstract of the article in the *Letters of JETP* [31] devoted to the determination of the anisotropic chemical shift ^{13}C in the NMR spectrum of hexamethylbenzene.

Another problem arose in the study by NMR of ^{27}Al nuclei with spin 5/2 and electric quadrupole moment. Quadrupole broadening as well as dipole broadening in the *first approximation of perturbation theory* could be eliminated by rotation at the same angle to the direction of the polarizing magnetic field as the dipole broadening, since the correction of the first approximation at the value of the electric quadrupole interaction (EQI) is proportional to the expression:

$$\nu_Q (1 - 3\cos^2\theta_l),$$

where ν_Q is the frequency of the nuclear quadrupole resonance of the nucleus under consideration in the absence of a magnetic field, θ_l is the angle between the directions of the main axis of the EQI tensor and the magnetic field. However, if EQI is not too small compared to the NMR frequency (Larmor frequency ν_L), the broadening will be determined *in the second approximation of perturbation theory* by an expression proportional to $(\nu_Q^2 / \nu_L) \cos^2\theta \cdot (1 - \cos^2\theta)$. If, nevertheless, EQI is not so large that it was necessary to take into account the following approximations of perturbation theory, proportional to (ν_Q^{n+1} / ν_L^n) , then a second rotation is suggested around the axis perpendicular to the field direction ($\cos^2\theta_2 = 0$) or directed along the field ($1 - \cos^2\theta_2 = 0$). One of the successful representatives of the Lippmaa school A. Samoson together with the outstanding American scientist A. Pines in 1988 developed a device for an NMR sensor with two rotors [33] that ensured the elimination of EQI broadening via *elimination of both the first and second order*, what is important for research in solid NMR of nuclei such as ^{14}N , ^{27}Al , etc. with moderate EQI values (the NQR frequency is much less than the Larmor frequency if magnetic field is strong enough).

In the late 1970s and in the 1980s, the Lippmaa's

laboratory developed cooperation with the chemists of the GDR (G. Engelhardt, (Berlin, Adlerhof) and others, see, for example [36]) and of the Czechoslovakia (Jan Shraml and others, see for example, [37]). Their joint work was devoted mainly to chemical radiospectroscopy of ^{29}Si NMR, primarily in solid silicates and in molecules containing trimethylsilyl groups. Separate articles were published in the Journal of the American Chemical Society, a significant number of publications with Czech chemists were published in English in the Czechoslovak journal and some works were published with German chemists in GDR.

In 1980, Endel Lippmaa together with G.V. Skrotzky edited a Russian-language translation of the monographs Haeberlen and Mehring combined in one edition [38, 39]. The subject of this publication [40] («High resolution NMR in a solid state») has found only limited application among Soviet specialists.

The Endel Lippmaa's successful researches in this area, was awarded the AMPERE's diploma and prize in 1994.

The most recent meeting between Endel Lippmaa and his Soviet colleagues took place in 1988 in the village of Viitna between Tallinn and Kohtla-Järve.

It was the Second Joint School for the Application of NMR in Chemistry and Petrochemistry gave a lecture on high-resolution NMR in solids. It was an extremely informative and understandable lecture for specialists. After the School, its participants visited the laboratory of Lippmaa in Tallinn and got acquainted with the new equipment.

Lippmaa's work in the field of ion cyclotron mass spectrometry

Let us dwell on the work of Lippmaa and his colleagues on high-precision measurements of the nuclide mass differences. The first attempts to master for this purpose a new method of ion-cyclotron resonance mass spectroscopy (ICR MS) were undertaken by Lippmaa in 1980. The method is based on the action of the Lorentz force in a magnetic field and resonant transitions from one closed orbit of motion to another under the action of an alternating electric field. The resonance frequency of the ICR ω_i in this case is equal to:

$$\omega_i = Hq_i/m_i,$$

where H is the magnetic field strength, q_i , m_i is the charge and mass of the ion i .

The ICR method with the Fourier transform [41] has developed in 1974. Lippmaa immediately under-

stood: the method is appropriate for implementation on a kind of modification of the NMR spectrometers with the Fourier transform. In 1984, using an ICR MS equipment with a field strength of 4.7 T (magnetic and computer system of a PMR spectrometer at 200 MHz could be a base for an ICR spectrometer) created with the support of company Bruker, Lippmaa and his colleagues performed an experiment to determine the mass difference of tritium $^3\text{H}^+$ and helium-3 $^3\text{He}^+$ ions (^3He nucleus is the product of β -decay of the tritium nucleus). This result (estimate of the upper mass limit of the neutrino (antineutrino $\tilde{\nu}_e$) m_{ν} , equal to $18588 \pm 3\text{eV}$ [42, 43]) is still cited in the world literature (at least 45 citations in the WoS database), although the model chosen for the evaluation was inappropriate. This episode in the scientific life of the Lippmaa's Laboratory was overshadowed by the fact that with the help of the same company (Bruker Spectrospin), researchers from the Institute of Chemical Physics of the Academy of Sciences of the USSR published the same results two months earlier [44]. However, the links go mainly to the article by Lippmaa et al. in journal Phys. Rev Letters [43]. In recent publications about the neutrino mass estimation, the interest in which was fueled by some cosmological hypotheses and the results of the mission of the spacecraft of the European Space Agency PLANK (2009 - 2013), for example [45], mentioned another model for estimating the upper limit m_{ν} . This is the mass difference in the pair of nuclides $^{187}\text{W} \rightarrow ^{187}\text{Re} + e^- + \tilde{\nu}_e$, which turned out to be equal to 2555 eV, that is, about an order of magnitude less. Based on some theoretical models of neutrino physics and data from other experimental methods, it is now believed that the neutrino mass is still many times smaller.

RESULTS OF THE SOVIET PERIOD

A result of twenty-eight years of successful work of Lippmaa in the field of chemistry and physics is very fruitful. Some data about Lippmaa collaboration with Soviet specialists see too in *Application* (memoires of Yu. A. Ustynyuk [46], with links to [47- 50] and our comments).

With the collapse of the USSR and the return of Estonia's independence, Lippmaa's scientific career was far from over, although he had to share his time and energy between political and scientific activity. The beginning of his political activities was initiated by the so-called «phosphate war», the struggle against the deployment of phosphate rock in northeastern Estonia. The direction of his further political activities was the

restoration of Estonia's independence. When Endel Lippmaa passed away, most of his obituaries (for example in [1]) began with a mention of his role in finding copies of the secret protocols to the Soviet-German Pact of 1939. I would like to give an excerpt from the memoirs of astronomer Jaan Einasto, Lippmaa's successor as academic secretary of the Physical Sciences Division of the Academy of Sciences of Estonia [51]: *«I would especially like to emphasize the contribution of Endel Lippmaa to the restoration of Estonian independence. Among the scientists, he was undoubtedly the most active fighter on the front line for the cause of Estonia. And he achieved success: he managed to succeed in the phosphate war (struggle to stop the development of phosphate rock in Estonia), and then, being a deputy of the Supreme Council, managed to squeeze opponents and make Moscow recognize the existence of <a secret protocol to> the Molotov-Ribbentrop Pact».*

Further scientific researches of Lippmaa and his disciples in the late 1980s, in the 1990s and after 2000 were devoted to the study of solids by NMR and molecular spectroscopy. Some of his works even before 1990 were devoted to the study of the structures of high-temperature superconductors by magnetic resonance methods. Lippmaa also collaborated with the European CERN Center in programs CMS (Compact Muon Solenoid) and TOTEM (TOTAl cross section, Elastic scattering and diffraction dissociation Measurement at the Large Hadron Collider).

A certain intermediate result of twenty-eight years of successful work of Lippmaa in the Soviet period should be summed up.

Firstly, Lippmaa created a national Estonian scientific school in the field of chemical NMR spectroscopy. It was one of the strongest scientific schools in this field in the former USSR. Let us note that the some other scientific schools enjoyed much more substantial material and organizational support by the scientific leadership of the USSR.

Secondly, Endel Lippmaa became involuntarily one of the leading experts in the NMR in the USSR. This imposed certain duties on him, with which, however, he coped without much difficulty, thanks to his outstanding erudition and organizational skills. What he resolutely avoided was everything connected with the problem of industrial production of NMR equipment in USSR, he was cooperating with German-Swiss company Bruker instead.

Thirdly, Lippmaa and his school received a number of scientific results of the world level. First of all, these are pioneering studies of ^{13}C and ^{15}N NMR spectroscopy of a number classes of organic compounds and original

research and development in the field of high-resolution NMR spectroscopy in solids. Lippmaa was awarded for the achievements in this field of spectroscopy by the AMPERE price, and one of the employees of Lippmaa Ago Samoson with his inventions was involved in the work of the instrument-making firm Bruker. A group of researchers in the field of ion-cyclotron resonance mass spectrometry (ICR MS) also appeared in the new Institute of Chemical and Biological Physics of the Academy of Sciences of the ESSR.

And for the Soviet specialists in NMR, the most important result of their long years of cooperation with Lippmaa was the possibility of sharing scientific knowledge with foreign colleagues. Thanks to the high authority of Endel Lippmaa in the scientific world and his very loyal attitude to Soviet colleagues, they had access to important international events that Lippmaa supervised, and to placing his publications in an international scientific journal, to whose editorial board he was a member. In [52], in the spirit of the Tallinn city jargon, this opportunity was called *«Lühike jalg to Europe»*. *«Lühike jalg»* literally means «short leg» in Estonian, that is, the shortest path (meaning the path from the center of Tallinn to the upper part of the city). In addition to the Soviet chemists with Lippmaa, as we mentioned above, the chemists of the GDR and Czechoslovakia cooperated with him fruitfully.

In 1994, in the midst of the «evil nineties» of Russia, the AMPERE Congress was held in Kazan, dedicated to the half-century anniversary of the discovery of the EPR by E.K. Zavoisky. For reasons we can only guess, Endel Lippmaa refused to come to this Congress, saying that he does not want to apply for a Russian visa. The star of this second in the territory of the former USSR Congress AMPERE was Richard Ernst (Swiss), a recent (1991) Nobel laureate in chemistry for the use of pulsed one-dimensional and multidimensional NMR spectra. The NMR technique was moving forward and double resonance methods, so successfully mastered by Lippmaa, were replaced by two-dimensional spectroscopy methods.

Analyzing his results and the history of their achievement, it can be noted that Endel Lippmaa, in part of NMR, was not a «discoverer of new paths», but undoubtedly was an outstanding pioneer of these paths. And it was very relevant in the XX century, the century of «big science». The study, dedicated to one of the prominent «founders» of the NMR pathway in chemistry, J. Roberts (USA) [53], contained the statement:

«During the 1960s, organic chemistry underwent a dramatic transformation as a result of the introduction of high-tech tools. In this process, nuclear magnetic

resonance (NMR) has become an important analytical method in organic chemistry.»

In a sense, Endel Lippmaa could claim in the scientific community of USSR chemists the same role that J. Roberts, J. Schoolery and similar researchers played in the United States. Of course, he recognized too the merits of VF Bystrov and others in solving this important task in the USSR. In addition, Lippmaa, in contrast to the above-mentioned scientists, did not stop in his work on analytical applications of NMR in solutions and picked up a high-resolution NMR development initiative in a solid state, and contributed to ion-cyclotron mass spectrometry.

Endel Lippmaa possessed all the necessary qualities for this role: engineering intuition, excellent knowledge of physics and chemistry, the sheer charisma of the leader. His fantastic working capacity and ability to manage his time caused surprise and admiration. But there was one more, extremely necessary quality: «the feeling of the possible». It is the talent of the born politician. This sense of the possible was needed just under the conditions of the «Wonderland» (of the Soviet reality and the complicated situation of Estonia in the Soviet system), as well as the rapid development of NMR technology abroad with the lagging of Soviet technology in this area.

At the same time, his interests extended far beyond the chemical applications and development of NMR technology. In more favorable conditions, Lippmaa's contribution to world science could be more meaningful. Undoubtedly, he was not satisfied with the conditions in which his scientific creativity developed. Perhaps in contact with other eminent scientists who worked in the same area of science abroad, he would have achieved even greater results, but such contacts were almost impossible for him. Doesn't the Ustynyuk's (see *Application*.) comparison between the erudition and the efficiency of Lippmaa and the Nobel laureate R. Hoffmann speak about this? However, (note in parentheses), the comparison of these two outstanding scientists is not quite legitimate. This is evidenced by the difference in their second specialties. Lippmaa is a famous politician, and Hoffmann is a brilliant writer and poet.

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Lippmaa's doctoral dissertation and provided significant assistance in choosing the place of publication, as well as in editing the article. I thank especially S.V. Semenov (Kurchatov Institute) for his analysis of the role of Lippmaa's work on ion-cyclotron resonance in neutrino physics. Author is grateful to GA Kessenikh (freelance teacher of mathematical methods in economics at Moscow State University), which completed a preliminary editing of the translation of the article from Russian into English.

APPLICATION. FROM PERSONAL MEMORIES OF PROFESSIONAL COLLABORATION WITH E.T. LIPPMAA

The author was lucky to have among his closest colleagues Yuri Alexandrovich Ustynyuk (see about him [46]) direct student of A.N. Nesmeyanov, an outstanding specialist in the field of organoelemental chemistry and high resolution NMR, who headed for a long time (1968–2012) interdepartmental NMR laboratory on chemical faculty of Lomonosov Moscow State University. Yuri Alexandrovich was closely associated by work and scientific interests with E.T. Lippmaa. Author in 1974 attended the defense of the doctoral dissertation Yu.A. Ustynyuk «Structure and intramolecular rearrangements of cyclopentadienyl compounds of the IVB group», where Lippmaa was one of the official opponents. Therefore, it was decided to turn to Yuri Alexandrovich with a request: to evaluate the text and the content of the proposed publication. With the permission of Yu.A. Ustynyuk we give here his memories, sent to the author by e-mail. Friendly criticism and additions to the initial outline of our article, which accompanied the mentioned memories, we, were possible, clarified and fully considered when finalizing the article. Our comments and additions in the text below are in italics.

Yuri Alexandrovich wrote about the following:

I have read everything carefully. You see, I had a very special and very complicated relationship with Endel. They began after he opposed my dissertation in 1974. Of course, he gave an excellent review, having understood more than two other opponents in his work (Academician OA Reutov in Chemistry and Adolf Bolutin (Quantum Calculation Specialist)). But at the same time, when I met him at the station, Lippmaa handed me a notebook on 24 pages, written in small handwriting, which contained comments and criticism on mistakes, inaccuracies, stylistic errors. I have never heard such a deep, comprehensive analysis. It turned out that Lippmaa brilliantly knew chemistry, catalysis, quantum

chemistry, and all spectroscopy. After defending my dissertation, for about a month I carefully studied everything that he wrote and sent him letters with explanations. He answered very carefully, and in controversial cases, gave a bunch of references to literature. In general, this correspondence has greatly changed my view of who a serious scientist is, how he should work.

I accepted the first invitation of Lippmaa to oppose a dissertation to someone from his team with great joy. Of course, I tried not to lose face and thoroughly worked the text. I also brought 8 pages of comments. At first there was a defense, and then a coffee seminar. After that, I was accepted in this company as my own, opposed several doctoral works, including in 1983 the work of Tönnis Pehk «NMR spectroscopy of ^{13}C isomeric aliphatic and carbocyclic compounds». Almost every year I received invitations to seminars in Tallinn at the very end of February (they were on Fridays), after which I then with the whole team on Saturday and Sunday skied in their camp, sat in a sauna. In turn, Endel, when he was in Moscow, usually visited us at the faculty for half a day. After lunch, different people gathered around him and threw questions at him. These visits caused great interest among people from other laboratories. They wrote to him, and on arrival he allotted time to many. I have been a constant witness to these conversations. Endel with the professor of physical chemistry spoke as an equal to him, but more erudite scientist. He acted as a mentor with specialists in catalysis, offering his own solutions to problems. When one of the biochemists came, Endel gave a brilliant lecture on the structure and mechanisms of action of proteolytic enzymes. One of his visits to us was interrupted by a docent from the department of organic catalysis, who considered him/herself an important specialist in zeolites. *As it is known*, Endel made a series of brilliant works on the structures of zeolites using silicon and aluminum NMR in a solid [47–49]. *One docent* with great aplomb began to state the opinion on the mechanism of catalysis on zeolites, relying, it seems, on the ideas of the beginning of the 1930s. A terrible situation for me! And I just cannot stop it. Finally, it was said: «Well, now you understand how they work?» I thought that now Endel would berate his opponent completely. However, he smiled gently and, *with a touch of regret*, said: «Yes, now very many people think so.»

I think that in the article about his work it would be necessary to describe in more detail how important were his results obtained by the NMR method for solving chemical and physical problems that were then in the spotlight. Then it becomes clear what he actually did.

In the 70s, the latest issues of foreign scientific journals in natural sciences could only be found in the Lenin

Library and in the scientific hall of the Library of Foreign Literature, where they were in the public domain. So I always went there on Sundays. Once, having arrived there at 10.30 with a desire to look through the latest JACS issues, I did not find them on the shelf. I noticed, Endel was sitting, who has taken all 10 issues. Of course, he greeted me in a friendly way, I sat down next to him and began to look through the issues that he had already processed. Apparently, he mastered speed reading techniques *in English*. I barely managed to look through one issue, but he in the same time - three. At the same time, he was very quickly writing something in a notebook in fine, even handwriting. By two o'clock in the afternoon all JACS's he worked. I got up three times and rested for 5 minutes. He sat without straightening. His notebook ended, and he asked me for a couple more sheets of note paper. At three afternoon we had a snack in a cafe opposite to broth pies, and he quickly rushed off somewhere.

Yury Konstantinovich Grishin in our laboratory did several works on NMR of mercury-199. In Tallinn, he reported these works a couple of times at a seminar. Already in 1992 much later than the declaration of independence of Estonia, Endel gave a response from the "third organization" to Grishin's doctoral thesis «Nuclear magnetic resonance of mercury-199. The nature of magnetic parameters and applications in the study of the structure and dynamics of organo-mercury compounds». As always, his review was the most informative.

As you correctly write, one of the first 60 MHz NMR spectrometers in Estonia was created on the basis of a Japanese magnet *with an induction of 2.35 T*. This magnet went to Tallinn because the JEOL device purchased by the MSU chemical faculty did not have enough funds allocated to Moscow State University, *and the Techsnabexport manager deleted the magnet from the MSU application (he believed they will do the magnet themselves)* and did a favor by them to the new Institute Cybernetics in Tallinn, the application of which in other conditions would hardly have been implemented. And in our basement turned out to be useless electronics. It stood there for 10 years, and then I gave it to Endel for some symbolic amount. It was useful to him in creating the ion-cyclotron resonance spectrometer, with which he made experiments to estimate the neutrino mass. This part of Lippmaa's activity was not at all reflected in your text.

In all my life I have met only two people of such tremendous capacity for work, such a deep, comprehensive erudition. The first Endel, the second - Roald Hoffman, winner of the Nobel Prize in Chemistry (*in 1981 for the generalized quantum theory of molecular and atomic col-*



Figure 4. Yuri Alexandrovich Ustynyuk. from an article in the “Bulletin of Moscow State University” (2017). [46]

lisions), see about him [50], with whom I worked closely for several years. Of course, it is difficult to compare Hoffman (a brilliant theorist who collaborated with many outstanding scientists such as William Nunn Lipscomb, Robert Burns Woodward and others) and Endel Lippmaa, a brilliant and very erudite organizer of independent engineering and experimental research. However, I would like Endel to be recognized not only as a specialist in NMR, but as a brilliant versatile scientist. He was a surprisingly charming modest man with a soft sense of humor, but at the same time with iron principles.

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