野副鉄男 Tetsuo Nozoe

Tetsuo Nozoe (1902−1996)

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In the evening of March 27, 1996, the authors visited Professor Tetsuo Nozoe in a hospital in Tokyo. His bedside was half covered by chemistry periodicals, domestic and foreign. He looked and sounded much better than we had feared, and, though hoarse in voice, he told us about his current research work and the proofreading of his review article on azulenequinone[449] as enthusiastically as usual. As we had doctor's permission to visit him for only 30 minutes, we became anxious that the long conversation might fatigue him, and after an hour, we reluctantly mentioned that we ought to leave him, but he held out his hand, gesturing for us to stay for "*just another five minutes!*" He then continued to talk about his chemistry for another 30 minutes.

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Only eight days after our visit, Professor Nozoe quietly passed away at 4:40 in the afternoon of April 4, just before his 94th birthday. He had ended his stormy life entirely dedicated to chemical science, like an old, big tree falling down. Despite his age, his death struck unbelievable surprise and enormous sadness into his collaborators, friends and students, who had seen him walking around in a lively manner, discussing chemistry tirelessly, to make them believe that he really was an "iron man" who never dies, as his name Tetsuo means in Japanese.

As an initiator of a totally new type of discipline — nonbenzenoid aromatic chemistry — and the greatest contributor to the modernization and internationalization of Japan's organic chemistry in the post-war period, his death was regarded as an end of an era.

Now that seven years have elapsed since he passed away, it is a great honor and pleasure for his students to be asked to write an obituary of Professor Nozoe and to describe his personal profile and his contribution to chemistry.

I. The Early Years (1902−1926)

Tetsuo Nozoe was born in Sendai on May 16, 1902, as the sixth son of Shigeichi and Toyo, who had eight sons and three daughters. Shigeichi came from the Nagasaki Prefecture. After graduating from the Law School of Tokyo Imperial University, Shigeichi started his career in Sendai as a lawyer and got involved in politics, serving as a chairman of the City council of Sendai and later running for a position in the National Diet (Japan's House of Councillors). Having been a devout Christian and enthusiastic about her children's education, his mother, Toyo, enrolled her three daughters at an American missionary school in Sendai and sent them to piano lessons, which was something new in those days in Japan.

Both parents wanted Tetsuo to become a medical doctor, but he was deeply influenced by his teacher at Sendai First Middle School, and took much interest in chemical experiments. It was one of his joys to set up and perform experiments on a simple laboratory bench at the corner of the storehouse in his parents' premises. This turned out to be his immediate motive for choosing a career to which he dedicated 80 years — his whole life. It is interesting to compare his exposure to chemistry to Professor R. B. Woodward who also developed his interest at a very early stage, reading the chemical literature, performing chemical experiments in his home, and then carrying his "hobby" all the way through his life.

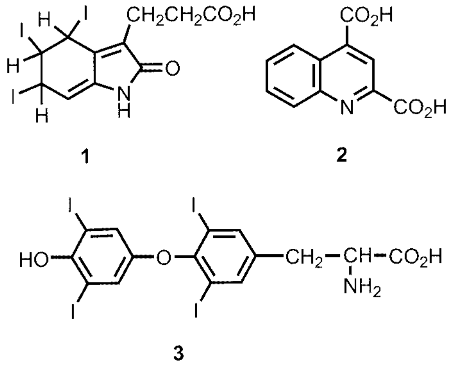
In 1919, Tetsuo proceeded to the premedical course at the Second High School in Sendai (there were only three high schools in Japan at that time), but found the chemistry courses there too descriptive and boring with no experimental demonstration. During this period of time Tetsuo developed an interest in western classical music, and enthused over a wide variety of music from symphony, concerto, to sonata and operas. Appreciation of music became his lifetime hobby and served as an oasis throughout his busy scholarly life.

Among Toyo's church friends was Masao Katayama, who was the physical chemistry professor of Tohoku Imperial University. He heard about Tetsuo's interest in organic chemistry and, without hesitation, strongly recommended that he enter Tohoku Imperial University where Professor Riko Majima (Japan's leading authority on organic chemistry, with a fine personality and excellent leadership) was teaching, although the school was only 15 years old. Tetsuo followed Professor Katayama's suggestion.

At that time in Japan, the national universities were called the "Imperial Universities"; Tohoku Imperial University was the third such university, after Tokyo and Kyoto. Although there were a few private universities, they offered literature and social science courses but no chemistry (or science). To satisfy the national needs, however, there were many colleges specializing in pharmacy, engineering, agriculture, and medicine, and they offered three-year courses after middle school. In addition, there were some women-only "universities" which also gave three-year courses in science after middle school. Being a young university, Tohoku had invented a quite revolutionary and democratic scheme to attract good students by giving a chance for the graduates of these specialized colleges and women's universities to take its entrance examination. As a result, many good students came by this route; about half of Tetsuo's 15 classmates came through this "irregular path". For the shy Tetsuo, who had spent a rather isolated childhood, this liberal atmosphere was a very good opportunity for making friends with classmates of various ages and backgrounds.

In 1925 (when in his 3rd year at the university), Tetsuo joined Professor Majima's group, to which he had been looking forward so eagerly, and started a research experiment. The project assigned to him by his mentor was entitled "Synthesis of Thyroxin, a Thyroid Hormone" (Scheme 1). The structure **1** had been proposed for this hormone by Kendall in 1920. To synthesize the compound Tetsuo started with the readily available quinoline-2,4-dicarboxylic acid (**2**) and carried out some reactions on its tetrahydro derivative. The result of this work was published by Nozoe himself in the *Proceedings of the Imperial Academy*[1] (in German) and in the *Journal of Chemical Society of Japan*[2] (in Japanese) and constituted his first original paper. Professor Majima gave Tetsuo no detailed instructions on or suggestions for the project, but encouraged him to pursue his studies independently with patience. This experience later proved to be extremely valuable.

Tetsuo also had a good fortune to be a member of Majima family, and to be acquainted with a group of excellent colleagues, who later played important roles in many phases and fields of organic chemistry in Japan. To name just a few, the following were the senior people when Tetsuo started working: Professor Chika Kuroda (the first female chemist in Japan, Tokyo Women's Normal College, Ochanomizu University), Professor Munio Kotake (Osaka University), Professor Harusada Suginome (President, Hokkaido University), and Professor Shiro Akabori (President, Osaka University). In addition, there were many talented and enthusiastic young people in Majima's group. The in-



Scheme 1. Some compounds mentioned in Nozoe's BSc thesis

teraction and fraternization with these members of the group and the stimulating atmosphere under which they worked was an indication of how important their research was to them; this must had had a tremendous impact on Tetsuo's later academic life.

In March 1926, Nozoe graduated from Tohoku University, and stayed there for a while as a sub-assistant in Majima's group. However, on the advice of his mentor, he soon departed Sendai for Formosa (later Taiwan), becoming a member of the Central Research Institute (Government Monopoly Bureau) in Taipei at the end of June. When he was leaving Sendai, Professor Majima came to send him off with a copy of a Biochemistry Journal, in order to show him another (correct) structure **3** for thyroxin, proposed by Harrison.

As it happened, Nozoe's hurried move to Formosa was arranged by Professor Majima. Initially frustrated, Nozoe was later deeply grateful for his mentor's warm consideration and thoughtfulness, when he later learned that Majima had made that quick decision because he thought that Nozoe should devote himself to his research work without getting involved in the troublesome political activities in which his father and younger brother were engaged.

Nozoe then stayed in Formosa for 22 years. When one thinks that this trip in 1926 was the very beginning of his long research life in the island, during which the foundation of today's nonbenzenoid aromatic chemistry was established (through the discovery of hinokitiol), one may be thrilled at the wonder of human destiny, but at the same time recognize the importance of fortitude in order to make the most of that destiny.

II. Research Activities in Formosa (Taiwan) (1926−1945)

As a subject, organic chemistry originated from the study of the chemistry of the compounds produced by living organisms. Therefore, it was natural that the main stream of organic chemistry in the early 20th century was concerned with the study of natural products. The research conducted by Nozoe was no exception.

His trip from Sendai to Taipei took five days by train and ship. It is obvious that Majima's advice had a great effect on Nozoe's decision to move to Formosa, but it is equally true that Nozoe thought that a tropical region like Formosa may have many interesting indigenous plants, providing him with the possibility of being able to find and work with interesting natural products. When he arrived in Taipei, it was over 35 °C and very humid, in contrast to Sendai in the northern part of Japan. This weather lasted for half a year. In 1926, the population of the island was four million, of which 190,000 were Japanese, 140,000 were of Polynesian extraction; the rest were Chinese. However, because almost all of the residents spoke Japanese (the official language) he had no linguistic difficulties in living or working there.

At this time, the Government Monopoly Bureau was manufacturing camphor from camphor oil, and therefore one of the most important jobs for the Research Institute was to find a way to utilize the byproduct. He started working under Dr. Kinzo Kafuku, the director of both the Camphor Research Laboratory and the Central Research Institute on this very project. He carried out the oxymercuration of linalool with mercuric acetate in the Camphor Research Laboratory, but soon moved to the newly established Industrial Organic Chemistry Laboratory and started investigations into the chemical constituents of taiwanhinoki (*Chamaecyparis obtusa* var. *formosana*, later changed to *C. taiwanensis*). As it turned out, this project led Nozoe to the discovery of hinokitiol and then to the opening up of the field of nonbenzenoid aromatic chemistry.

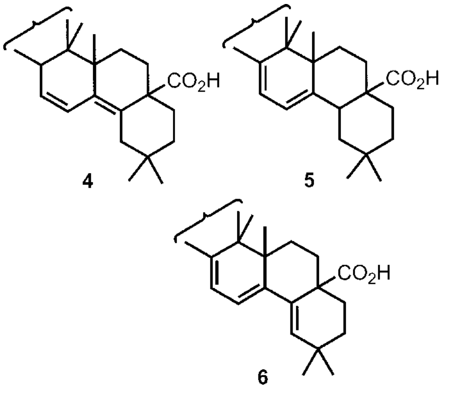
Taiwanhinoki was regarded as a variety of hinoki (*Chamaecyparis obtusa*), found widely in Japan. In Formosa there is another species called benihi (*C. formosensis*) whose constituents were investigated in the same laboratory. Both Chamaecyparis species in Formosa looked very similar to each other and were taxonomically indistinguishable; thus the plant materials obtained were often a mixture of both plants. Consequently, Nozoe had to go personally to the Arisan area (where these trees grow wild) in order to collect the leaves or wood chips from a single tree and then steam-distilled them. However, the investigation was finally suspended due to the difficulty in obtaining the genuine starting materials.

Kafuku, his boss, did not give him any detailed instructions whatsoever on the research, saying, "*You have to do your research work all by yourself. However, as long as you consider yourself a genuine pure chemist, you should never forget to display the originality in your research.*" Nozoe recalled the situation affectionately with the words, "*It was very useful for young chemist like me at that time to be initiated by deeply research oriented life in Professor Majima's laboratory and then to learn the importance of originality. I became fully aware of my responsibility to hew out a career for myself.*" He also described his fortune in being in the remote rural area and establishing friendly relationships with the leaders of organic chemistry in those days, in particular Professors Yasuhiko Asahina, Heizaburo Kondo and Eiji Ochiai when they visited Formosa on various occasions.

In 1928, the year Taihoku Imperial University was inaugurated, Dr. Kafuku became a professor, and following year Nozoe was promoted to the position of associate professor in the Faculty of Science and Agriculture. In the 13 years that followed, he worked on three major projects; Saponins and sapogenins (1929−1942), Wool wax (1936−1942), and Hinokitin and Hinokitiol (1935−1942).

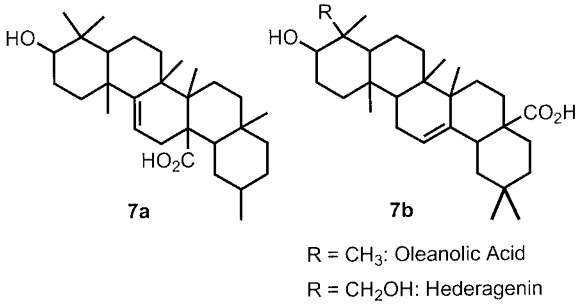
1. Saponins and Sapogenins

Soon after Nozoe resumed his own research in the Imperial University, Kafuku gave him some curious-looking fruits that he had collected in virgin forest at the southern tip of Formosa, and said, "*As they look like the legs of Japanese checkerboard, it might contain exotic compounds. Won't you check the components?*" With these words, Nozoe started his studies on saponins and sapogenins. The fruits collected by Kafuku turned out to be those of *Barringtonia asiatica* and *B. racemosa*. Although Nozoe isolated several polyoxygenated sapogenins by hard work (including the frequent collection of the plant materials by himself), the structures of the compounds were too difficult to be elucidated in those days, especially under his circumstances. However, he also investigated *Schima superba*, *Camellia japonica*, *C. sasanqua*, *C. sinensis*, *Glycine max*, *Styrax japonicus*, *Aesculus hippocastanum*, and *Gleditsonia japonica.* Realizing that, on the one hand, structural study of polyoxygenated sapogenols was not feasible, and stimulated by the pioneering work of Professor Ruzicka on amyrins, oleanolic acid, and hederagenin on the other hand, Nozoe then turned to the less oxygenated, simpler members of the triterpenoid family. By this time, a structure **7a** for oleanolic acid had been proposed by Ruzicka. Nozoe carried out the known oxidation and bromination reactions on oleanolic acid and hederagenin, obtaining the dehydro compounds **4** and **5** in addition to the known ketolactones and ketoacids. Bromination and dehydrobromination of **4** then yielded a trienecarboxylic acid **6** (Scheme 2). These three products exhibited absorption maxima at 245 nm, 290 nm, and 310 nm, respectively.



Scheme 2. Partial structures for the oxidation products of oleanolic acid and hederagenin

On the basis of this data, in 1937 Nozoe proposed the planar structure **7b** for oleanolic acid and hederagenin;[22−24] a slight modification of Ruzucka's old structure **7a** (Scheme 3). This structure was also arrived at later by Ruzicka and proven to be correct by numerous experiments that followed. This important achievement by the young Nozoe (and his few collaborators) clearly exhibits his remarkable flexibility and positive way of thinking. The measurement of the UV spectra alone was a tedious job and had never before been applied to the structure elucidation of colorless natural products, except for some plant pigments. Furthermore, his studies were performed under the inferior research conditions prevalent in Formosa in those days.



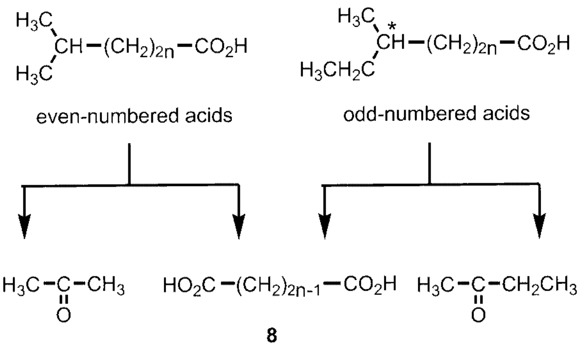
Scheme 3. Structures of oleanolic acid and hederagenin

2. Wool Wax

Wool wax, the secretion from sebaceous glands of sheep, can be obtained in quantity from the processing of wool, and therefore many studies have been conducted. At the time, wool wax was known to contain a mixture of unidentified acids and a large amount of unsaponifiable matter, consisting of cholesterol, triterpenoids, and wax alcohol. The structures of the triterpenoids, named lanosterol and agnosterol, were investigated by Windaus and Wieland, but after a long endeavor, they withdrew from this line of research. Nozoe was interested in the fact that triterpenoids, which usually occur in plants, were found in this secretion, and also in the inconsistencies in the reported experimental results. He therefore started to look into the constituents of wool wax.

Raw material provided by factories supportive of Nozoe's work was shipped to Formosa. The lanosterol and agnosterol obtained from separation of the neutral part of the mixture were subjected to chemical analysis. The reactions of lanosterol suggested to Nozoe that it was tetracyclic rather than pentacyclic, as had been proposed earlier. UV spectroscopy also played a crucial role here, in the assignment of chromophores in the various oxidation products. The tetracyclic nature of lanosterol as it stands today was confirmed by Barton in 1952.

As to the acidic part of the hydrolysates, solid structural evidence was lacking in the literature. After separating the mixture of the C_{14-20} carboxylic acids as their methyl esters by repeated fractional distillation (using a Widmer column), Nozoe plotted their $[\alpha]_D$ values against their carbon numbers, and observed a regular pattern. Further thorough fractionation revealed that (i) the crude acidic extract was a mixture of monocarboxylic acids with odd and even number of carbon atoms, (ii) all of the odd-numbered carboxylic acids were dextrorotatory whereas the even-numbered members exhibited no optical rotation, (iii) the even-numbered acids were not identical with any of the straight-chain C_{14-20} carboxylic acids, and (iv) the C_{26} and C_{30} straight-chain fatty acids were present. Oxidation of the even-numbered acids yielded acetone (together with the ω-dicarboxylic acids **8** with three fewer carbon atoms), whereas 2-butanone was obtained from the odd-numbered acids (Scheme 4). This led Nozoe to the unprecedented structures of the (optically inactive) lanolinic acid series and the (optically active) agnolinic acid series of carboxylic acids. He also identified many ω-hydroxycarboxylic acids, and showed lanocelinic acid to be ω-hydroxydotriacontanoic acid, $HO(CH_2)_{31}CO_2H$.



Scheme 4. Structural determination of the even- and odd-numbered fatty acids isolated from wool wax

Intrigued by the acidic components of wool wax, Nozoe then went on to investigate the acidic constituents of the fat from the sebaceous glands of various other mammals. He collected the hair of Mongolian camels, Japanese horses, and Japanese men, women, and children. However, he could only identify cholesterol and cholestanol, which was rather disappointing. He also collected the face fat of young Japanese females with the courtesy of Kao company, and obtained several grams of raw fat, but all of the samples and the records were lost during the move of the laboratory and bombing at the end of World War II.

Nozoe's enthusiasm also extended to the study of an insect called mengachu (*Aphis sternorrhyncha*), which is a pest of sugar cane and has a white silky coat. By an ether extraction of the body, he obtained colorless fibers which upon hydrolysis produced stearic acid, lauric acid, dodecanedioic acid, and glycerol. Thus the insect was wearing a nylon-like glycerol polyester! It is regrettable that these studies were discontinued because of World War II, the results only having been reported orally under the title of "The Difference in Body Fat and Skin Fat of Animals and Plants" at a meeting in 1942.

3. Hinokitiol

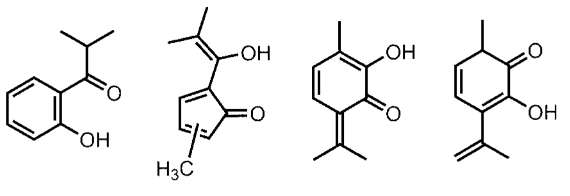
In 1935, Nozoe resumed his investigations into the constituents of taiwanhinoki — in particular the acidic constituents. The essential oil of the plant had already been studied by Uchida (1916), Tsuchihashi and Tasaki (1922), and also by Hirao, who was Nozoe's senior at Tohoku Imperial University. Hirao had isolated dark red crystals (m.p. 251 °C) from the steam distillation residue and reported it as hinokitin, $C_{30}H_{34}O_{10}$, in the *Journal of Chemical Society of Japan (Nippon Kagaku Kaishi)*. In 1926, he also reported the presence of a new acid, called henpakusan, $C_{10}H_{16}O_2$, which gave hinokitin on oxidation with ferric chloride.

Nozoe also isolated hinokitin during the isolation of the acidic components, and took the opportunity to perform some experiments. He observed the formation of ferric hydroxide on treatment of an ether solution with alkali, and isolated from the ether solution a phenolic compound, $C_{10}H_{12}O_2$, which regenerated hinokitin on treatment with ferric chloride. He named this colorless phenolic compound hinokitiol,[17] and concluded that hinokitin was a ferric complex of hinokitiol, $(C_{10}H_{11}O_2)_3Fe$. The presence in nature of an iron-containing pigment, its volatility (hinokitin sublimes in vacuo) and solubility toward organic solvents were beyond the imagination of organic chemists at the time (in the days before ferrocene). Thus Hirao's unawareness of the presence of iron is understandable.

In 1936, the *Journal of Chemical Society of Japan* planned to publish a special issue to celebrate Professor Majima's 60th birthday. Nozoe dedicated a paper on hinokitiol[17] to express his deep appreciation to his mentor, who had been warmly supporting him all the time. He was not able to present a structure, but simply described hinokitiol as a monocyclic α- or β-enolone with an isopropyl group, able to form complexes with almost any metal. This short paper was to be the first significant publication on his life's work.

The low hinokitiol content of taiwanhinoki (0.1−0.2 %) was the major obstacle impeding the progress of the research. However, this problem was solved by demand for the oil from another quarter. Professor Shigehiro Katsura had been newly appointed to the Faculty of Medicine at Taihoku Imperial University, having moved from Tohoku Imperial University. He was engaged in research into the application of capric acid and other fatty acids to the treatment of tuberculosis. Because these acids had become hard to secure because of the war, he came to Nozoe and asked for a substitute. Nozoe gave him a small amount of hinokitiol and (−)-rhodinic acid (which was also isolated from the acidic part of hinoki oil). Both compounds turned out to be significantly active against tubercle bacillus and some other bacteria.[25,27] With this positive result, the Takasago Perfumery Company, which had a factory in Formosa, decided to set up a plant to isolate rhodinic acid from taiwanhinoki oil. Therefore Nozoe was able to get hold of enough hinokitiol for his research. He also had a long standing interest in many other constituents of taiwanhinoki and benihi oils other than hinokitiol. This interest came to fruition in his time at Tohoku University (vide infra).

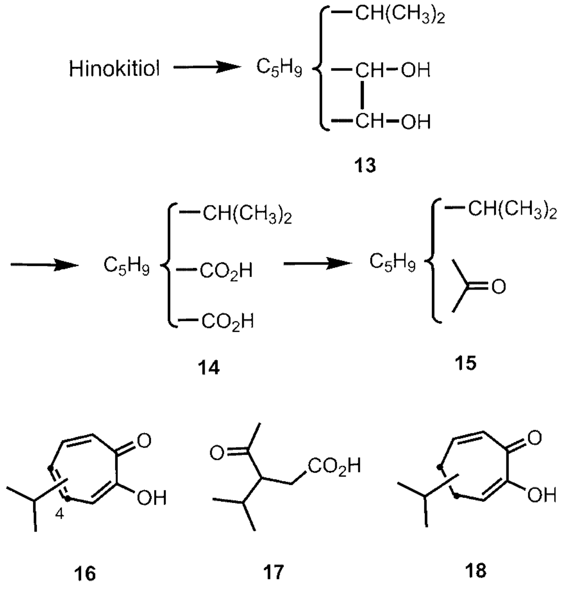
Now that Nozoe had sufficient hinokitiol, he was able to perform further investigations to elucidate its structure. The fact that hinokitiol provides acetone upon rigorous oxidation, together with the observation that it forms complexes with virtually all metallic ions initially led Nozoe to believe it had the *o*-hydroxyacetophenone structure **9** (Scheme 5). Because the metal complexes of hinokitiol have quite different properties from that of *o*-hydroxyacetophenone, however, he also considered the five-membered cyclic β-diketone **10** and the six-membered cyclic α-diketones **11** and **12** as possibilities.



9 **10** **11** **12**

Scheme 5. Some proposed structures for hinokitiol

However, none of these formulae satisfactorily explained the following observations: (i) hinokitiol is stable toward acids and alkalis, either being recovered unchanged or forming a salt, but it does not undergo converison to a catechol derivative, and (ii) on catalytic hydrogenation, hinokitiol gives the saturated monocyclic diol **13** ($C_{10}H_{20}O_2$), which can be oxidized to a dicarboxylic acid **14** ($C_{10}H_{18}O_4$), whose pyrolysis yields a cyclic ketone **15** ($C_9H_{16}O$). This last experiment clearly demonstrated hinokitiol to be a seven-membered cyclic α-enolone **16** with an isopropyl group (Scheme 6).

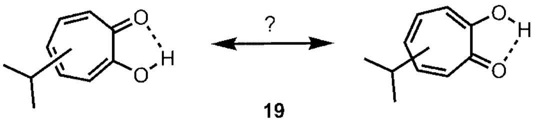


Scheme 6. Confirmation of the presence of the seven-membered ring in hinokitiol

Determination of the location of the isopropyl group was not easy, because some oxidation reactions resulted in almost complete destruction of the ring, whereas the reactions using metal-containing oxidizing agents led to the formation either of metal complexes or tar. The only reaction which gave a positive result was oxidation with alkaline hydrogen peroxide, from which β-isopropyllevulinic acid (**17**) and a dicarboxylic acid of unknown structure were obtained. This result unfortunately led Nozoe to ignore his elemental analysis result and propose the structure **18**,[25,26] containing two additional hydrogen atoms, instead of the correct structure **16** ($C_{10}H_{12}O_2$) (Scheme 6). This structure was reported at the annual meeting of Pharmaceutical Society of Japan held in Tokyo in April, 1940. Around that time, Professor Iinuma, who had carried out a study on the formation of complexes by hinokitiol (using the material provided by Nozoe), also reported the same incorrect structure **18** in his paper (1943). The paper had attracted the attention of Professor Erdtman in Sweden, and led to a lifelong intimate friendship between him and Nozoe (vide infra).

After giving his report in Tokyo, Nozoe hurried back to Formosa and started reexamination, and soon was convinced that the structure of hinokitiol must be **16** and not **18**, mainly from the elemental analyses of hinokitiol, its acetate, and its methyl ether. However, he suffered from long nightmares trying to explain the following unusual facts: (i) Hinokitiol dissolves in concentrated sulfuric acid and is recovered intact upon neutralization, (ii) the sodium salt of hinokitiol is stable in hot alkaline solution, and (iii) the acetate and methyl ether of hinokitiol are hydrolyzed exceptionally easily.

Because of the war, European chemical journals and books stopped arriving in Formosa in 1937, followed in 1941 by American books and journals, forcing Japanese scientists to be completely isolated from the rest of the world. Given these circumstances, it was good luck that Nozoe managed to obtain Pauling's "The Nature of Chemical Bond" (1939), probably one of the last scientific books to be imported into Japan before World War II. Reading the book, he arrived at the idea that hinokitiol might contain an entirely new type of resonance system **19** involving an intramolecular hydrogen bond (Scheme 7). This idea was introduced at a meeting in Formosa in 1941, but the reaction of the audience was cool.



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Scheme 7. A suggested explanation for the stability of hinokitiol

Towards the critical stage of World War II, research work (such as structural studies) was interrupted. Ironically enough, however, the large scale extraction of hinoki oil was encouraged by the military, in order to make up for the gasoline shortage. Because the military wanted only the neutral part of the oil, the acidic constituents were therefore left behind. Nozoe must have welcomed the situation with wild joy after the war was over, when he saw the dark red cake of hinokitin being discarded from the huge steel pot used in the extraction.

In the later stages of the war, it was virtually impossible to pursue fundamental research, and therefore Nozoe was forced to do war-time research. He took up an additional position as the head of the Chemistry Section in the Research Institute on Tropical Medicine (affiliated to Taihoku Imperial University), and worked in collaboration with Professor Katsura. They found that hinokitiol had significant activity against tetanus bacilli.[25,27] Because of the continuous heavy bombing in Taipei after 1942, the university had to evacuate the campus. Nozoe decided to move his whole laboratory. Therefore, all the members of his group disassembled and moved all the laboratory facilities (including benches with power lines, water and gas pipelines) to a mountainside about 20 km from the city, where, in a short time they built a new laboratory. However, on the very day they completed the work and everybody was ready to resume research, the war ended (August 15, 1945). They then had to repeat the process in the opposite direction to re-establish the laboratory back in its original location. It took a year to complete the work this time.

After World War II, Taihoku Imperial University (Japan) was renamed Taiwan National University (Republic of China). Nozoe and many other faculty members and technicians were asked by the Chinese government to stay and help with the reorganization of the university. He accepted the invitation and tried hard to establish his research project and reorganize organic chemistry in Taiwan. At the time, the many Chinese government officials who came from the mainland were educated in Japan, spoke Japanese very fluently, were civil to the defeated nationals, and treated them very generously.

The newly appointed Dean of the Faculty of Science at Taiwan National University, Dr. Su Bu-Chin (a graduate of Tohoku Imperial University and a mathematician) delivered an impressive opening speech of the Faculty before the faculty members and students, saying, "*The reason why Taiwan has developed today is mainly due to the continuous effort of the people living here, that is, you people, and also due to the guidance of Japanese people. Being responsible to the education in Taiwan, and therefore to Taiwan in future, you should bear this in your mind and try your best in the future research work.*"

Right after the war, Nozoe had recovered the distillation residue of hinoki oil (consisting mostly of hinokitin), from the Taipei Factory of the Takasago Perfumery Company with the permission of Chinese Army, who had been suspicious about his request for the dirty-looking reddish "mud" deposited in the corner of the factory. This material gave about 10 kg of pure hinokitiol. With this Nozoe was ready to go.

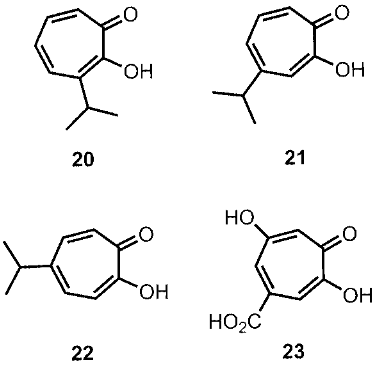
As was mentioned briefly before, he regarded hinokitiol as not just a natural product, but a compound with a novel aromatic system, and he therefore concentrated his efforts on achieving electrophilic substitution. Indeed, hinokitiol was found to undergo substitution reactions.[34] Treatment with bromine gave mono-, di-, and trisubstituted products. Nitration did not take place under strongly acidic conditions, but treatment with nitric acid gave a yellow dinitro compound, which aromatized to methyl 6-isopropyl-3,5-dinitrobenzoate upon recrystallization from methanol.[31] This experiment confirmed both the position of the isopropyl group and the *ortho*- (3- and 7-positions) and *para*- (5-position) -directing effect observed in the electrophilic substitution of the hinokitiol system. Hinokitiol also underwent azo coupling to give orange azo dyes,[33] but on recrystallization from alcohols, reddish-purple crystals were formed.[65] Observing such unusual behavior, Nozoe was convinced that hinokitiol had the classical structure **16** (with the isopropyl group at the 4-position). However, his final conclusions on the structure of this interesting compound had to wait until his return to Japan in 1948.

III. Post-War Research at Tohoku University (1948−1966)

1. The Dawn of Troponoid Chemistry

Although the research at Taiwan National University was quite active and productive, and against the opinions of his many Chinese colleagues and students, Nozoe left Taiwan at the end of May 1948, responding to the invitation of his alma mater, Tohoku University (renamed as a part of the post-war change of the entire educational system in Japan). He took up the position of lecturer in June and was later promoted to a professorship, succeeding Professor Hiroshi Nomura in December.

Because of the food shortage in Japan in those days, many Japanese people leaving Taiwan to return home did so carrying food and other necessities; sugar was the most popular item. However, Nozoe did not follow the general trend; he carried on his shoulder chemistry books, thermometers, graduated cylinders, perforated porcelain discs for vacuum filtration, 500 mL each of benzene and chloroform and, above all, a large quantity of hinokitiol! Of course, this needed the special permission of the Chinese authorities. This attitude was characteristic of Nozoe, who thought only about his research interests, and tried to continue his work in the hardship of the post-war period in Japan.



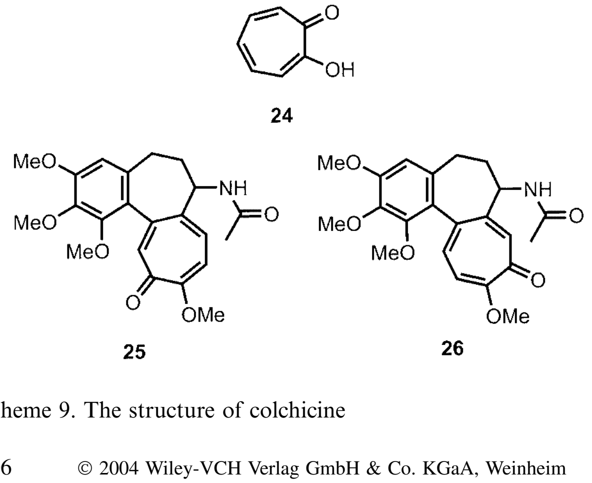
Scheme 8. Some natural products containing the tropolone ring

At the end of 1948, Nozoe received a letter from Professor Shigehiko Sugasawa at the Faculty of Pharmacy in the University of Tokyo, containing a copy of Professor Holger Erdtman's letter from Sweden and a reprint of his paper on the structure of thujaplicins published in *Nature* in that year. Erdtman had isolated three isomeric monoterpenoids from *Thuja plicata* (named the α-, β-, and γ-thujaplicins) and had shown them to have the structures **20**, **21**, and **22**, respectively (Scheme 8).

Professor Erdtman had written to Professor Sugasawa because they were very close friends, having spent their early years together at Professor R. Robinson's laboratory in Cambridge. While studying thujaplicins, Erdtman had found Iinuma's 1943 paper on the metal complexes of hinokitiol (vide supra) in *Chemical Abstracts* (1947), and had realized that somebody in Japan had isolated and was conducting research on a seven-membered cyclic compound having very similar properties to his thujaplicins.

Erdtman and Nozoe continued their correspondence and found hinokitiol to be identical to β-thujaplicin (which is now the more commonly used name), but more importantly established a lifelong friendship. Professor Holger Erdtman's wife, Professor Gunhild Aulin-Erdtman, started her memorial letter as follows: "*Tetsuo Nozoe was born on May 16, 1902, when Holger Erdtman was 20 days old. They grew up and started their career completely unaware of each other, separated by geographic, cultural, and warfare barriers. It was to take 47 years, deep interest in science, and a helpful friend to break the mighty communication barriers.*"[450] She also described the wonderfulness of their encounter and the depth of their intimate friendship.

In his paper, Erdtman mentioned the structure of stipitatic acid (**23**) (Scheme 8). This structure was proposed by Dewar (*Nature*, 1945) based on the careful analysis of Raistrick's experimental results. At the same time, Dewar proposed the name "tropolone" for the basic structure **24**. In this paper, he also proposed the structure **25** (containing the tropolone ring, later revised to **26** by Šantavý) for the alkaloid colchicine, which is obtained from *Colchicum* species and exhibits interesting physiological properties (Scheme 9). The structural analysis was again entirely based on the experimental results obtained by Windaus and Cook.



Scheme 9. The structure of colchicine

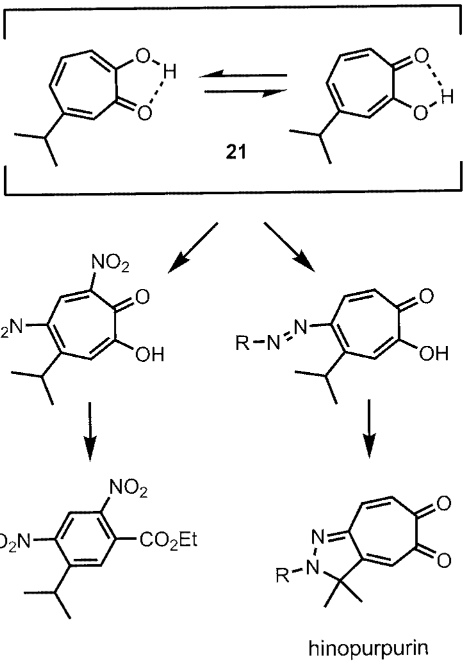
In the 1940s, it was impossible to subscribe to foreign journals in Japan; only in 1951 was Nozoe able to obtain Dewar's paper and realize that active research on natural tropolones was currently in progress around the world. However, the lack of information was not limited to Japan. Professor E. Heilbronner described his situation in his memorial to Professor Nozoe,[450] "*In his biography, Professor Nozoe tells us that one of the major handicaps from which his research suffered during World War II was the lack of foreign periodicals, with the result that he remained uninformed about work abroad. However, the reverse was also true, and it was thus a considerable surprise to learn at the beginning of the 1950s, that much of what we and others had planned to do in the field of non-alternant systems, especially of tropones and tropolones, had already been achieved with spectacular success in Formosa (later Taiwan) by someone called Tetsuo Nozoe.*"

Chemical research on a natural product is generally complete when it is isolated, its structure determined, and a rational synthesis achieved. However, Nozoe was convinced of the existence of novel aromatic character in hinokitiol because of its unusual stability towards acids and alkalis, and its ability to undergo facile electrophilic substitution despite its high degree of unsaturation. He therefore focused his activity on the synthesis of tropolone as well as hinokitiol, in order to aid future investigation of their chemical and physico-chemical properties as well as their biochemical and pharmacological behaviors.

In 1949, Nozoe wrote an article entitled "Studies on Hinokitiol" in a journal called *Yakugaku (Pharmaceutical Science)*.[404] This is a summary of his lectures at Osaka and Tokyo Universities on the subject of his results obtained in Formosa. In this article, he gave the structure of hinokitiol as 4-isopropyltropolone (**21**), rationalized a variety of reactions that hinokitiol and its derivatives underwent, and suggested the future extension of the work to the physico-chemical investigation of tropolone and the chemistry of azulene, heteroazulenes and other seven-membered and five-membered unsaturated cycles. This article is a masterpiece and even today discloses Nozoe's deep foresight into the emerging field of the chemistry of novel aromatic strucures.

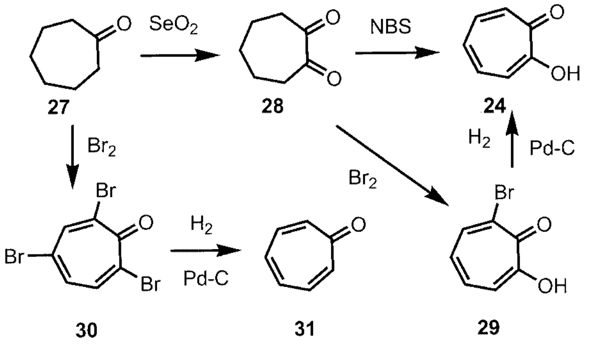
Although Nozoe was convinced that hinokitiol was 4-isopropyltropolone in 1941, his convictions were only published in an academic journal in 1949.[404] He was therefore only the third person to propose the tropolone structure for a natural product, after Dewar's work on stipitatic acid and colchicine in 1945, and Erdtman's suggested structures for the thujaplicines in 1948. Nevertheless, Nozoe was a remarkable man, as is clearly shown by his ingenuity in recognizing hinokitiol as a novel aromatic compound, and by his extraordinary enthusiasm and exceptional stamina in pursuing his ideas.

At Tohoku University, Nozoe resumed his studies on the reactions of hinokitiol and its derivatives, with special regard to the substitution pattern in the electrophilic substitution of the natural product. He converted some of these compounds (including hinokitiol itself) into the corresponding benzoic acid derivatives (as shown by the example in Scheme 10), either via their methyl ethers or by conventional functional group conversion. He also observed an interesting oxidative cyclization[65] that occurred on heating arylazohinokitiols (obtained by azo coupling of hinokitiol) in polar solvents. Furthermore, it was established that hinokitiol (**21**) existed not as a resonance hybrid, but as a pair of tautomers that interconverted through intramolecular hydrogen bonding (Scheme 10).



Scheme 10. The tautomerism of hinokitiol and some of its substitution reactions

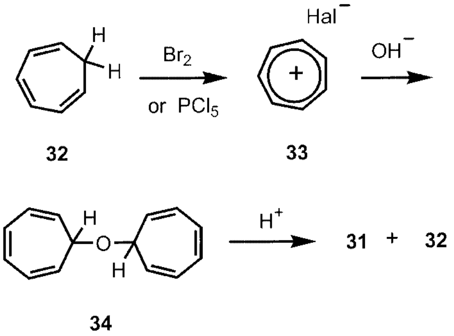
Encouraged by this success, he started on the synthesis of tropolone (Scheme 11) as well as hinokitiol and its isomers. In his synthesis of tropolone, cycloheptanone (**27**) was oxidized to the corresponding dione **28** by SeO_2. Whereas treatment of **28** with NBS gave a low yield of tropolone (**24**),[28] treatment of **28** with bromine induced both bromination and subsequent dehydrobromination, to give 3-bromotropolone (**29**), which, on catalytic hydrogenation, afforded tropolone. Hinokitiol,[29] α-thujaplicin,[30] γ-thujaplicin,[54] and methyltropolones[50] were synthesized by a similar route. The results of these studies were mostly reported in *The Proceedings of Japan Academy* in 1950 and the years that followed. The choice of the journal reflected the difficulties (including shortage of paper) in publishing academic articles in Japan in those days. Nozoe was very fortunate to have Professor Majima, his mentor and supporter, as a member of the Japan Academy. Recognizing the significance of Nozoe's work, Professor Majima offered to explain his results to his fellow academy members once a month, to be published in the *Proceedings* in the following month. In this way alone, Nozoe published 79 papers between 1950 and 1954.



Scheme 11. Syntheses of tropolone and tropone

The synthesis of tropolone turned out to be an unexpected international competition; four communications appeared nearly simultaneously, one from the USA (Doering, in *J. Am. Chem. Soc.*, accepted April 4, 1950), two from the UK (Cook, in *Chem. Ind.*, accepted June 3, 1950, and Haworth, in *Chem. Ind.*, accepted June 10, 1950), in addition to Nozoe's (in *Proc. Jpn. Acad.*, accepted April 2, 1950).[28]

The synthesis of tropone (**31**), the parent ketone, was accomplished both by Dauben (1951) and Doering (1951). Nozoe also synthesized[47] tropone in 1952 by the controlled hydrogenation of the tribromotropone **30**, which in turn was formed by the exhaustive bromination of cycloheptanone (**27**) (Scheme 11). In 1954, the positively charged tropylium ion was synthesized by Doering by the bromination of tropilidene (**32**), and was found to be identical with the compound obtained in 1891 by Merling using the same reaction sequence. The tropylium ion is the fundamental heptagonal carbocyclic system, and contains 6π-electrons, in common with both the cyclopentadienide ion and benzene, and led to the recognition of Hückel's rule proposed in 1932. Doering (1954) and Volpin (1957) had both synthesized ditropyl ether (**34**) by the treatment of tropylium halides **33** with alkali; in 1960, Nozoe developed a new synthesis of tropone (**31**) by the acid-induced disproportionation of this ether[164] (Scheme 12).



Scheme 12. Formation of tropylium halides and their conversion into tropone

In around 1950, scientific journals such as the *Journal of the American Chemical Society,* the *Journal of the Chemical Society*, and *Nature* became accessible to Japanese chemists. It became obvious that British chemists were particularly interested in tropolone chemistry, and consequently the Chemical Society organized a symposium on "Tropolone and Allied Compounds" in November 1950. Professor Erdtman made a comment and disclosed that "*the man who discovered tropolone was not me but a Japanese chemist called Tetsuo Nozoe.*" Therefore, his name became known among chemists all over the world. Professor Erdtman's fair attitude toward Nozoe has also been described in a Japanese primary school text book.

Learning the lively research activities going on in the western world, Nozoe felt quite strongly the necessity of communicating his research results to the world in more direct way. So he sent a review article (of about 20 pages) outlining his research in Formosa and Sendai, along with reprints of his papers, to Professor J. W. Cook, the chairman of the above symposium. Cook responded promptly, saying that he had never realized that so much in this field had been studied in Japan, and in 1951 kindly forwarded his entire manuscript to *Nature* as a special contribution.[58] Lord Todd also made arrangement through Professor A. W. Johnson to add a long comment on the research in Japan as a footnote in *Science Progress* he was summarizing. The result of Nozoe's research was thus published through the courtesy of his potential British competitors. We sense Nozoe's deep appreciation in his words "*I was deeply impressed by their attitude and the respect that they showed us*".[447] In the middle of his period of devotion to troponoid chemistry, he was awarded the Asahi Cultural Award in 1952, and the Japan Academy Award in 1953 on his "Study on Hinokitiol and the Related Compounds".

In 1953, Nozoe took a long leave of absence (May 15 to October 29) and went abroad for the first time in his life. The prime purpose of the trip was to attend the 14th IUPAC Congress, deliver a lecture entitled "Recent Advances in Troponoid Chemistry" and visit chemists all over the world to discuss topics of mutual interest. In his first stop (Darmstadt, Germany), he was warmly received by Professor and Mrs. Schöpf. On parting, Nozoe produced a note book and asked them to write a comment. Thus, they became the first to write a comment with their signatures in Nozoe's autograph book. Since then, he always carried the note book everywhere he went, and collected over 4000 signatures and comments from famous chemists all over the world. Many chemists outlined or depicted their research at the time of the writing, and the books thus represent an interesting profile of post-war chemistry. These nine volumes (shown in Figure 1) are now kept in the Tohoku University Archives, along with other items related to Professor Nozoe.

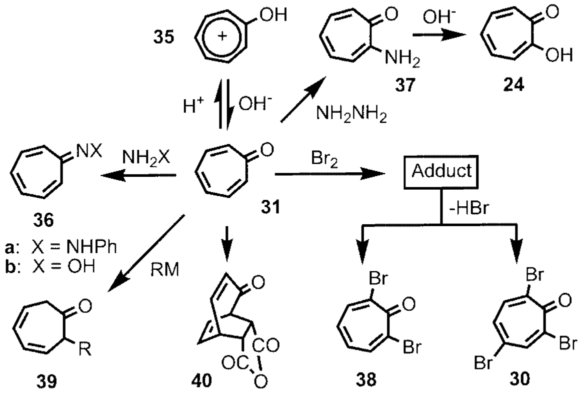
In his trip, which lasted for over five months, Nozoe visited more than 120 universities and research institutes in Germany, Sweden, Norway, Denmark, Finland, Switzerland, France, the UK, and the USA, and delivered a total of 26 lectures.

2. The Chemistry of Tropone and Tropolone

After the completion of the syntheses of tropone and tropolone, Nozoe concentrated his efforts on clarifying the chemical reactivity of tropone and tropolone as well as of hinokitiol. Consequently it soon became clear that (i) although unstable towards alkali, tropone (**31**) is stable toward acids by virtue of the formation of hydroxytropylium salts **35**, (ii) tropone exhibits carbonyl character to a certain extent, and (iii) it behaves as an unsaturated compound. For example, tropone reacts with phenylhydrazine or hydroxylamine to give the phenylhydrazone **36a** and the oxime **36b**,[97] but with hydrazine it unexpectedly forms 2-aminotropone (**37**), alkaline hydrolysis of which yields tropolone (**24**), representing a convenient and efficient path from tropilidene to tropolone (see Scheme 12). The action of bromine

Figure 1. Nozoe's autograph books

on tropone results in the formation of an addition product, which undergoes gradual dehydrobromination to give a mixture of 2,7-dibromotropone (**38**) and 2,4,7-tribromotropone (**30**).[98] Grignard reagents and alkyllithiums yield the adduct **39**,[173] and maleic anhydride gives the [4+2] adduct **40**[161] (Scheme 13).



Scheme 13. Some reactions of tropone

On the other hand, tropolone (**24**) exists as a mixture of tautomers, which interconvert by means of intramolecular hydrogen bonding. The derived acetate **41** also exists as a mixture of tautomers through 1,9-acetyl migration[257] (Scheme 14). In contrast to tropone (**31**), tropolone (**24**) is stable toward alkali and gives salts or complexes **42** with various metallic ions but, like tropone, it does form the stable 1,2-dihydroxytropylium ion (**43**) with acids. Because of the basicity of tropolone (**24**), electrophilic substitution reactions do not take place under strongly acidic conditions, such as those required for Friedel−Crafts alkylation and acylation. Sulfonation occurs with sulfamic acid,[37] but not with concentrated sulfuric acid. With various other electrophilic reagents, it forms substitution products **46**. The directing effect of tropolone in these reactions is as follows. Nitrosation and azo coupling occur exclusively at the 5-position, nitration[63] and sulfonation[45] preferentially at the 5-position, halogenations preferentially at the 3- and 7-positions and hydroxymethylation exclusively at the 3(7)-position.[75] Hydroxylation by peracid occurs both at the 3- and 5-positions.[112] Reduction of the arylazo or nitroso compounds affords 5-aminotropolone,[51,73] from which 5-hydroxytropolone can be derived. 5-Nitrosotropolone (**47**)[62] exists in the tautomeric diketo form and gives the corresponding trioxime and the quinoxaline derivative **48**. It also reacts with two moles of arylamine to give a rearrangement product **49**[326] (see Scheme 14). Numerous tropolone derivatives can be obtained from these substitution products.

Although the carbonyl group in tropolone is rather unreactive, the hydroxy group can easily be converted into a halo, methoxy or tosyloxy group, and these groups in turn can regenerate the hydroxy group upon hydrolysis. Thus, the tropolone system can be considered to be a vinylogous carboxylic acid, and, in fact, the acidity of tropolone falls between that of carboxylic acids and phenols.[61] These derivatives, called "active troponoids", undergo various nucleophilic reactions. The reaction pathway depends on the active troponoids, the nucleophilic reagents employed as well as the reaction conditions. The substitution pattern was clarified by using specifically labeled tropolone, hinokitiol and other homologues. The initial attack of nucleophile can occur either at the 2- or 7-positions, giving positional isomers in some cases, or at the 1- or 3-position to give benzenoid products. The following is a crude generalization. 2-Methoxytropone (**50**) is in most cases attacked at C-2 to give the "normal" substitution products, 2-halotropones **51** give "abnormal" substitution products (through attack at C-7) and aromatization products (through attack at C-1), and 2-tosyloxytropone (**52**) reacts at both C-2 and C-7 to give both the "normal" and "abnormal" substitution products. Utilizing an "abnormal" reaction, the conversion of γ-dolabrin to β-dolabrin was accomplished[160] (Scheme 15).

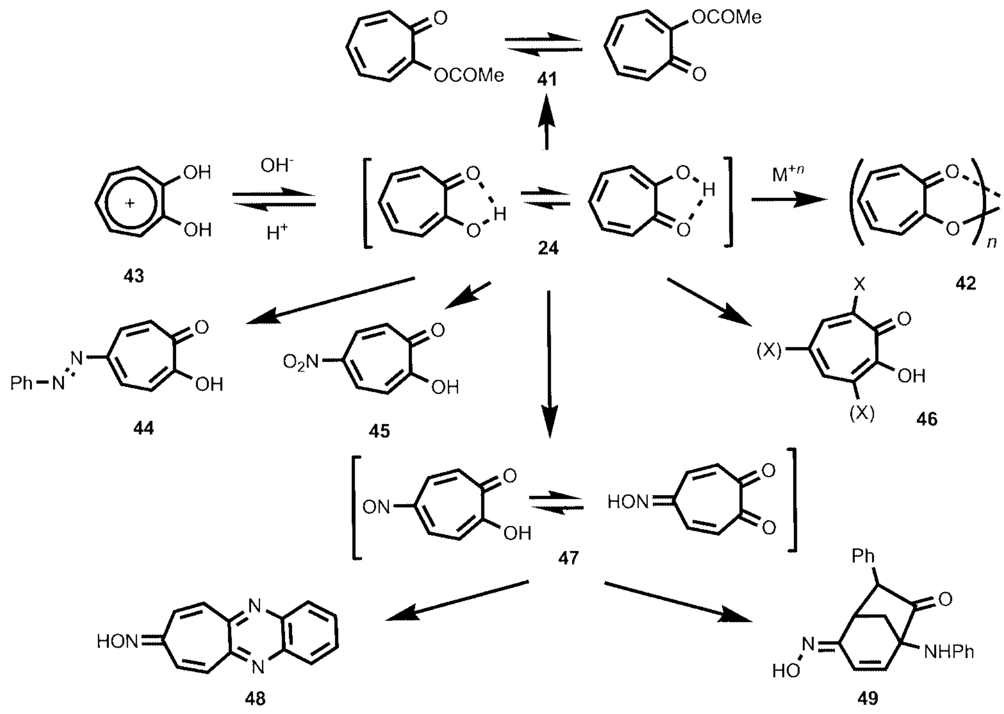
Based on his firm belief that the troponoids (and azulenoids described in the next section) were aromatic compounds, Nozoe felt it necessary to expand the research on these compounds from organic chemistry into physicochemistry, in order to elucidate the fine details of their structure. The following areas were all investigated in collaboration using material provided by Nozoe: Determination of dipole moments,[40,52,53,60,110,111,167] magnetic susceptibilities,[118,135] ionization potentials[154] and dissociation constants,[61,189] quadrupole resonance, near infrared, Raman, and UV absorption spectroscopy, metal complexation,[183] electron diffraction and X-ray crystallography studies, molecular orbital calculations, mass spectrometry,[198] and polarography.[136,132,155]

Through the meetings held from time to time, a deeper understanding of the physical properties of the troponoids and azulenoids was achieved, and, as a result, these compounds gained a firm footing in aromatic chemistry.

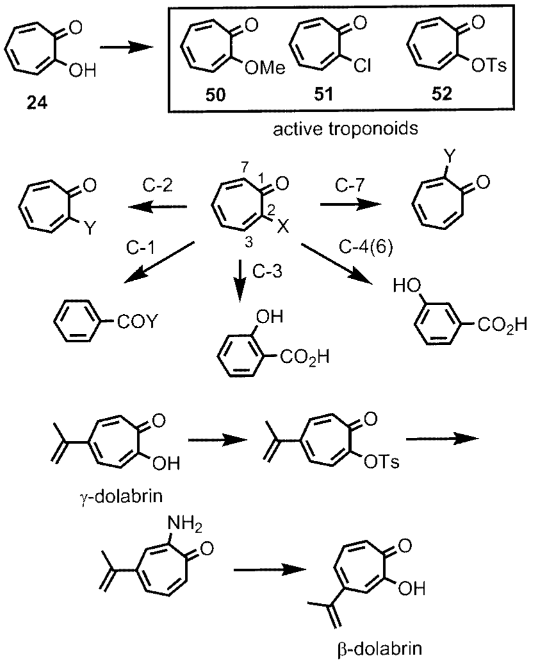
3. The Conversion of Troponoids into Azulenoids

Since the seven-membered ring system in troponoids is also incorporated in the azulene system, Nozoe launched studies into the conversion of troponoids to azulenoids immediately after his return to Sendai in 1948. Starting from "active troponoids", he developed efficient syntheses of many heteroazulenes and azulene analogs, such as 1-azazulenes **53**,[132,133] 1,3-diazazulenes **54**,[115,120,125] 1,2,3-triazazulene (**55**),[122] the derivatives of 1-oxazulanones **56**,[103] and 1-thia-3-azazulanone **57**[176] (Scheme 16).

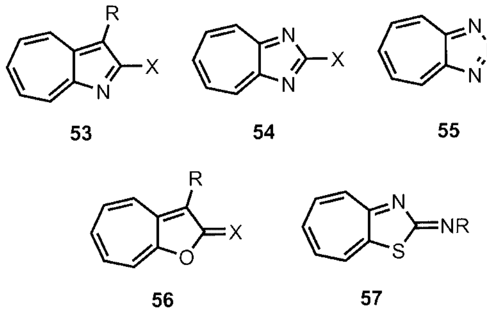
In 1953, he extended the nucleophilic substitution reaction of active troponoids to carbon nucleophiles; 2-chlorotropone (**51**) was treated with ethyl cyanoacetate in the hope of obtaining an oxa analogue of azulene **56** (X = O, R = CN), $C_{10}H_5O_2N$. In practice, however, two compounds **58** ($C_{16}H_{17}O_4N$, orange prisms, 70 % yield) and **59** ($C_{14}H_{11}O_3N$, orange needles, trace amount) were obtained[141] (Scheme 17). An amino group and a conjugated ester were detected in **58**, and a hydroxy group, a conjugated cyano group, and conjugated ester detected in **59**. This beautiful demonstration of the power of IR spec-



Scheme 14. Some reactions of tropolone



Scheme 15. Formation of "active troponoids" and their nucleophilic substitution products

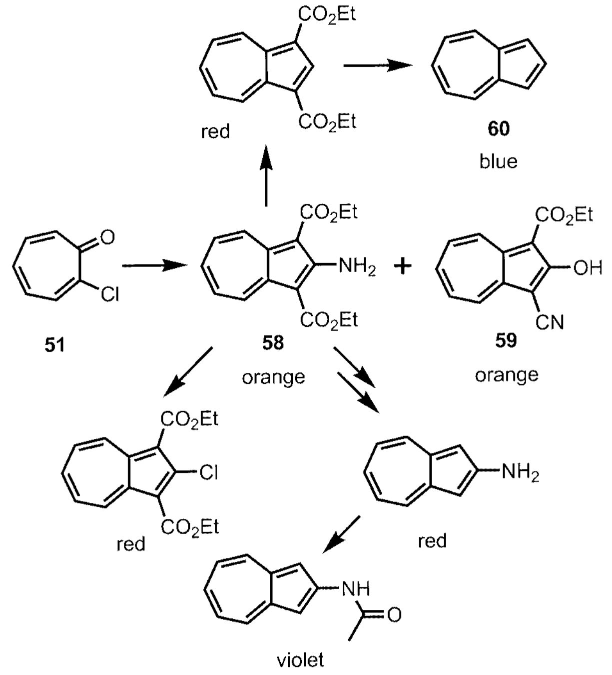


Scheme 16. Heteroazulenes derived from troponoids

troscopy for functional group detection (especially for **59**, the minor product), had a strong impact on Nozoe, who immediately realized the importance to organic chemists of free access to UV, IR, MS, and other spectroscopic methods. Whereas hydrolysis and subsequent thermal decarboxylation of the main product **58** yielded an isomer of naphthylamine as red needles (the acetate of which was a beautiful violet), the hydrochloride of **58**, on reaction with isoamyl nitrite, gave red crystals ($C_{16}H_{15}O_4Cl$) after a dramatic color change of the solution. Similarly, the deamination of **58**, followed by hydrolysis and decarboxylation, afforded the beautiful blue azulene (**60**)[141,145,213] (Scheme 17).

This unexpected, but very smooth conversion of troponoids to azulenoids provided easy access to a variety of substituted azulenes. Previously, synthesis of substituted azulenes (especially of those having substituents on the five-membered ring) had been almost impossible, because the classical synthesis involved the construction of a saturated carbon skeleton and thermal dehydrogenation at temperatures as high as 300−400 °C. Nozoe was very excited by this finding and talked about it affectionately throughout his life whenever he had a chance.

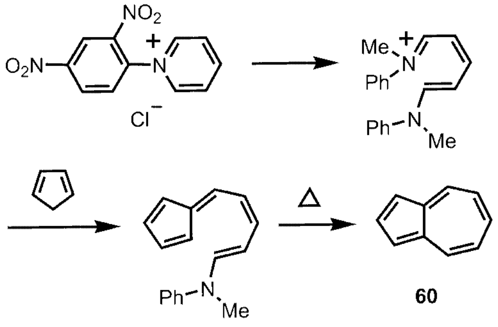
In 1955, Ziegler and Hafner developed another important, short and efficient synthesis of azulene (**60**), starting from cyclopentadiene and the Zincke salt derived from pyri-



Scheme 17. Synthesis of azulenes from active troponoids

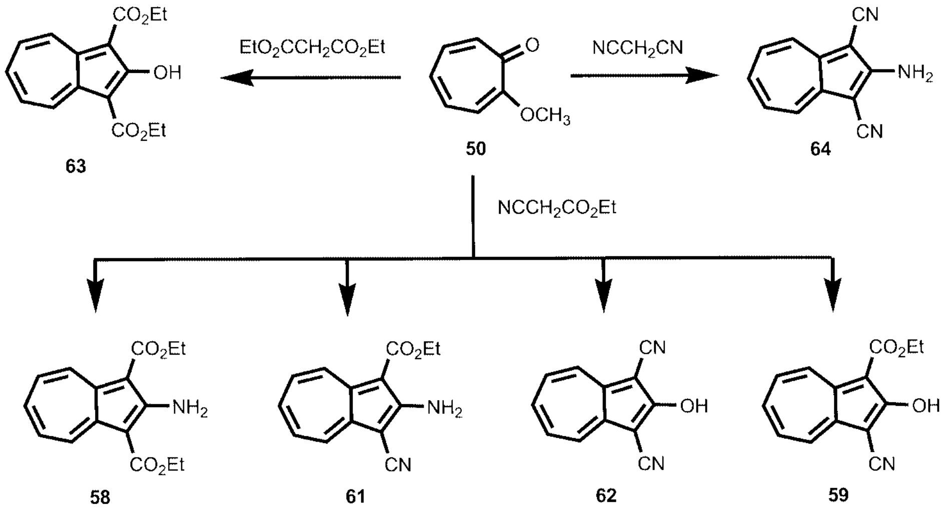
dine (Scheme 18). This method, later improved by Hafner, is an important azulene synthesis and complements Nozoe's method.

Continuing his efforts, Nozoe expanded his azulene synthesis to start from various "active troponoids". Thus, using two equivalents of ethyl cyanoacetate, ethyl malonate, or malononitrile, he was able to obtain variously functionalized azulenes (with groups at the 1-, 2-, and 3-positions) in a single step. Scheme 19 illustrates the reaction of 2-methoxytropone (**50**) with the compounds mentioned above. In the reaction of ethyl cyanoacetate, as many as four azulene derivatives (**58**, **59**, **61**, and **62**) were formed, whereas with ethyl malonate and malononitrile, **63** and **64**, respectively, were formed both in excellent yield.[145]



Scheme 18. The Ziegler−Hafner azulene synthesis

At the end of June 1957, Nozoe took a four-month leave of absence, and set out on his second trip around the world. The prime purpose this time was to deliver a plenary lecture entitled "Chemistry of Natural Tropolones and Allied Compounds"[414] at the IUPAC Congress in Paris, held to commemorate the centenary of the French Chemical Society. During this trip, he visited more than 100 universities and research institutes in Europe and USA, gave more than 40 lectures, and exchanged chemical information with both old and new friends. Professor K. Hafner affectionately recalls those early days and his first impressions of Nozoe as follows: "*In the summer of 1957, I met Professor Nozoe for the first time at the occasion of the 16th International Congress of Pure and Applied Chemistry in Paris, where he presented a most exciting plenary lecture. He also kindly attended my short contribution on 'Synthesis and Reactions of Azulenes' at the meeting. Immediately afterwards, he contacted me and involved me in a long and stimulating discussion on the azulene synthesis developed recently by him and his co-workers as well as by us. I admired his beautiful samples, accurately packed in cigarette cases, and I was deeply impressed by his high chemical standards and considerable interest in research, accompanied by a striking personal warmth. This was the beginning of a unique relationship with him, lasting almost forty years. At the same time, he kindly*



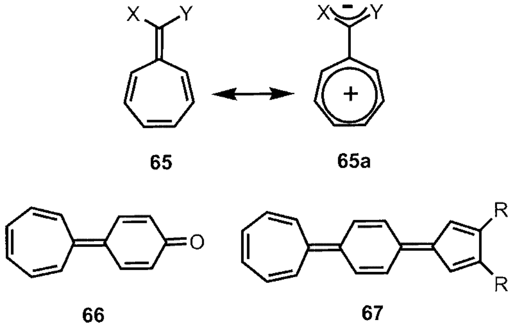
Scheme 19. Synthesis of substituted azulenes from 2-methoxytropone

accepted my invitation for a one day visit of my laboratory at the University of Marburg/Lahn, subsequent to his stay in Paris. This day was for me, as a young scientist, a great event, which I still remember with gratitude and pleasure."[450]

In November 1958, Nozoe was awarded the Order of Cultural Merit, the highest honor for a Japanese scientist, and became concurrently "A Man of Cultural Merit". In 1959, he became an honorary citizen of Sendai, his home town and the location of Tohoku University.

4. Expansion to the Heptafulvene System

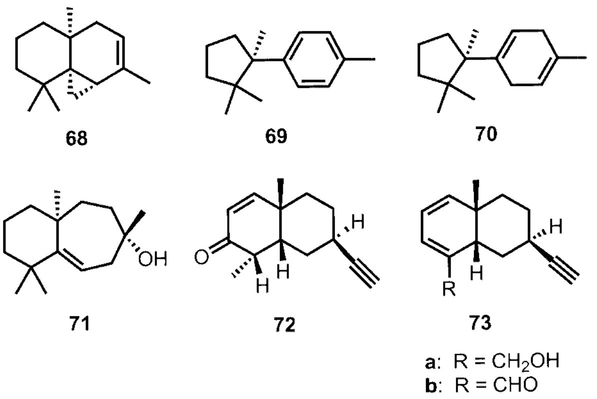
Heptafulvene **65** (X = Y = H), corresponding to a methylene homolog of tropone, had attracted the attention of chemists from a theoretical point of view, and was first synthesized in 1960 by Doering. The compound is too unstable to be isolated, but it was predicted that the system would be stabilized by the introduction of electron-withdrawing group(s) at either or both of the X and Y positions. Indeed, Nozoe synthesized and isolated 8,8-dicyanoheptafulvene (**65**, X = Y = CN) as stable orange crystals.[180] Furthermore, from its dipole moment, he showed that the ground state is more accurately represented by **65a**.[167] Subsequently, he successfully synthesized two extended conjugated quinoidal systems, benzoquinonetropid (**66**)[222] and the quinalenes **67**[264] (Scheme 20). The seven-membered rings in these compounds were also shown to be positively charged.



Scheme 20. Heptafulvenes and their extended analogues

5. The Neutral Constituents of Hiba and Benihi Oils

The Hiba tree (*Thujopsis dolabrata* var. *hondae*), grown in the Aomori Prefecture in the northern part of Honshu, Japan, had became known to contain considerable quantities of hinokitiol. Nozoe had carried out the investigation of the chemical constituents of the tree in connection with his aim of securing large amounts of hinokitiol. During this investigation, he had isolated a new tropolone called β-dolabrin[152] and had established its structure as 4-isopropenyltropolone (vide supra). He found that the Hiba tree contained 1 % each of hinokitiol and β-dolabrin, making the tree the richest source of terpenic tropolones in the world. He also extended his research to the neutral extracts, which resulted in the isolation and structure determination of thujopsene (**68**),[158] cuparene (**69**),[159] cuprenene (**70**),[159] and widdrol (**71**)[178] (Scheme 21). The benihi tree (a conifer indigenous to Taiwan and one of his interests at the beginning of his career) also received his attention. In 1963, he obtained the essential oil, and, after fractionation, isolated three norsesquiterpenoids named chamaecynone,[226] dehydrochamaecynenol, and dehydrochamecynenal.[241] Their structures were determined in due course as **72**, **73a**, and **73b**, respectively, and all were synthesized[274] (Scheme 21). Interestingly enough, these compounds have their ethynyl group in an axial orientation, with nonsteroidal *cis*-decalin conformations.



Scheme 21. The neutral constituents of Hiba and Benihi

In May 1965, Nozoe reached 63, the age of mandatory retirement at Tohoku University. Therefore, in April 1966, he became an emeritus professor at Tohoku University.

IV. Research Activities after Retirement (1966–1996)

As an unwritten law of national universities in Japan, retired professors are not allowed to stay and work on the university campus. Nozoe's biggest concern was therefore to secure the space to carry out his research. After some struggles with the government, he secured an office and a laboratory on a temporary basis. Two years later, the Kao Company provided him with an independent laboratory in their Tokyo Research Institute, named the Nozoe Research Laboratory. This consisted of an office and a laboratory and was supplied with personnel. Thus Nozoe reestablished connections with chemists in several companies (including the Kao Company and the Takasago Perfumery Company), as well as various national and private universities, and was able to continue with his research, right up until the end of his life (at that time still another 28 years away). Throughout, he always tried very hard to disseminate knowledge of troponoid chemistry.

In addition to his famous autograph book, Nozoe always carried with him a notebook, in which he described every aspect of his research, including extensions of ongoing research projects, future projects, problems or difficulties and their possible solutions, mechanisms of his reactions, results of discussions, and opinions of his colleagues and collaborators. A page from one of his 50 notebooks (which are now kept with his family) is shown in Figure 2.

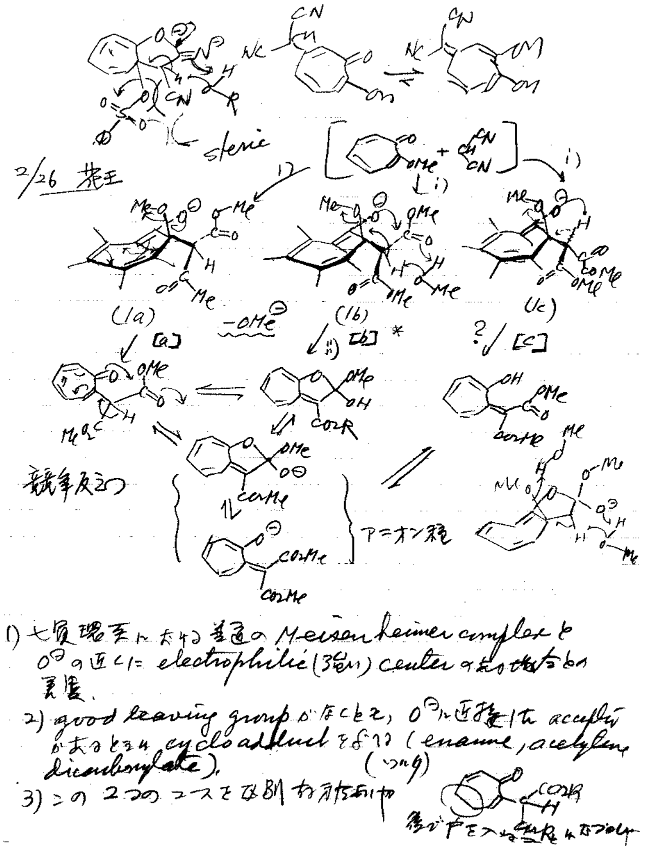


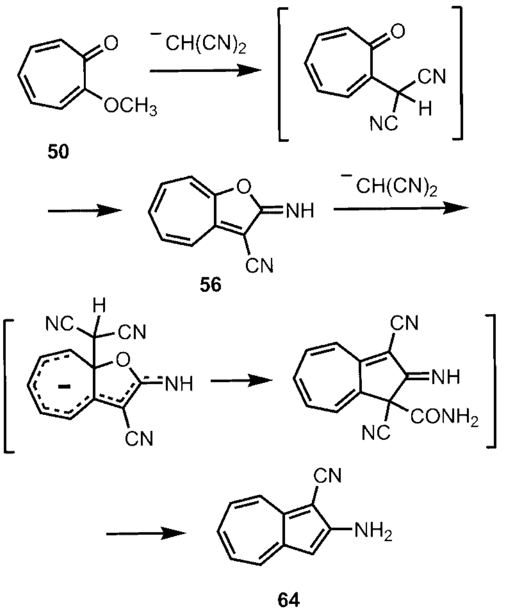
Figure 2. A page from one of Nozoe's notebooks

A story which vividly illustrated Nozoe's continued enthusiasm towards his research work was told by his widow after his death. Being completely absorbed in his research even after his retirement from Tohoku University, Nozoe used to make telephone calls or send facsimiles to his collaborators whenever an idea came to him, even if it was midnight. Unable to be indifferent, she reproved him, saying, "*It must be a nuisance to the person to receive the call at this late hour.*" His reply was "*The man can't pursue a research project if he is already in bed at this time of the day.*"

1. The Synthesis of Azulenoids from Troponoids; Extension and Reaction Mechanisms

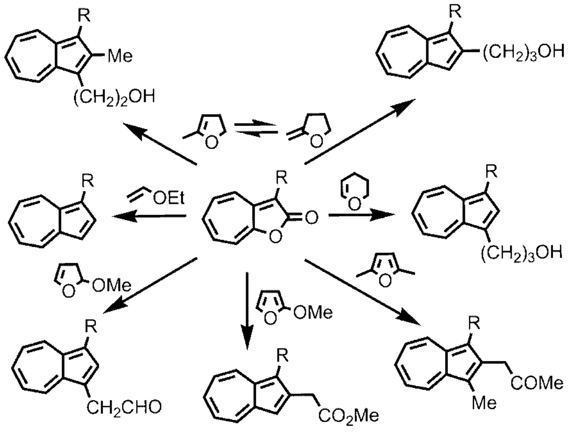
Nozoe had predicted the presence of intermediate oxazulanone derivatives in the pathway of his one-step azulene synthesis described above. His studies in this area were directed towards accumulating experimental evidence for the rationalization of reaction mechanisms and the design of new azulene syntheses.

The product obtained from the reaction of 2-methoxytropone (**50**) with one equivalent of malononitrile was proved, as expected, to be an oxazulanimine **56**. This product was treated with ethyl malonate in different solvents in the presence of various bases at different temperatures. The reactions were thoroughly monitored, and indicated that a remarkable number of reversible and irreversible reactions were in competition with each other. Nozoe identified the 13 intermediates, and also followed the fate of these intermediates under the above reaction conditions; they were all transformed into the final azulenic products. These mechanistic studies vividly disclose Nozoe's intense curiosity and inexhaustible spirit of inquiry. The reaction of 2-methoxytropone with two equivalents of malononitrile is summarized in Scheme 22.



Scheme 22. The mechanism for the formation of 1,3-dicyano-2-aminoazulene

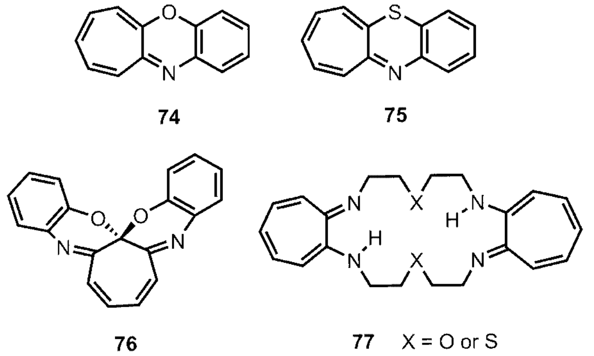
Nozoe also accomplished the synthesis of a variety of azulenes using various vinyl ethers as shown in Scheme 23.[320,325,342] This method was complementary to the enamine route developed by his former student Takase, at Tohoku University.



Scheme 23. Synthesis of various azulenes from 2-oxazulanone

2. Troponoids Condensed with Heterocycles

At Tohoku University, Nozoe had already conducted extensive studies into heteroazulenes and other seven-membered rings fused with thiophene, pyridine, and quinoxaline. He had also found that "active troponoids", on treatment with *o*-aminophenol or *o*-aminobenzenethiol, gave cycloheptabenzoxazine (**74**)[270,271,273,280] and cycloheptabenzothiazine (**75**),[168,287] respectively. At the Nozoe Laboratory in Tokyo, he extended his research into this area, and found that complex systems, including the compound **76**,[286,295] could be synthesized. He also prepared a compound **77**, which he named tropocoronand, and demonstrated its potential for the chelation of ions[277,289,291] (Scheme 24).



Scheme 24. Troponoids condensed with heterocycles

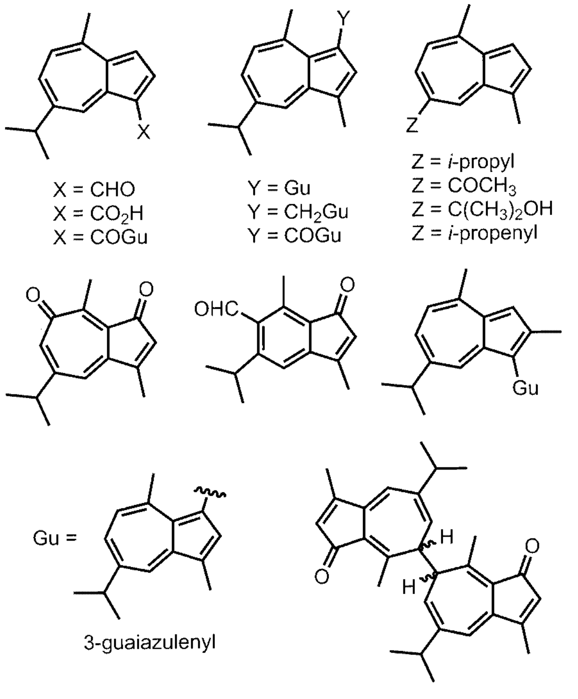
Professor F. Bickelhaupt describes his impression of Nozoe at the time as follows: "*In 1981, Professor Nozoe came back to Amsterdam and gave a sweeping lecture at the Vrije Universiteit on 'Synthesis and Reactions of Cyclohepta[b][1,4]benzoxzine', and his audience was duely impressed, among others, by the perseverance and enthusiasm with which he was able to prepare and to separate reaction mixtures containing approximately 65 products!*"[450]

3. The Autoxidation of Azulene

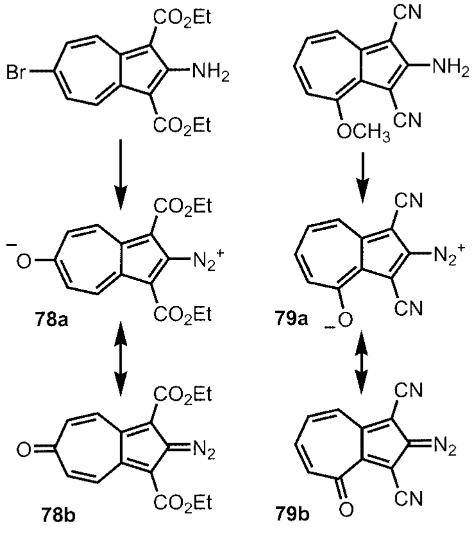
It had been known that azulenes were slowly oxidized during storage, although they were believed to be stable in air. At the age of 90, Nozoe challenged the structure elucidation of the oxidation products. Solid azulene was either spread on filter papers and exposed to air for extended periods, or oxidized by peracid, and the products obtained were separated by HPLC, and the structures established by physicochemical methods. Some of the products resulting from the heating of guaiazulene at 100 °C for 24 hours in dimethylformamide are shown in Scheme 25.[448]

4. Azulenoquinones

The isolation of a quinone from the above oxidation of guiazulene indicated the stability of azulenequinones. Further confirmation came from his own work on the diazotization of 6-bromo- and 4-methoxy-1,3-disubstituted-2-aminoazulenes. The products of these reactions were the unprecedented diazonioazulenequinolates **78a** and **79a**, respectively, both of which exist as resonance hybrids with the corresponding diazoazulenequinones **78b** and **79b**[262,358,376] (Scheme 26).

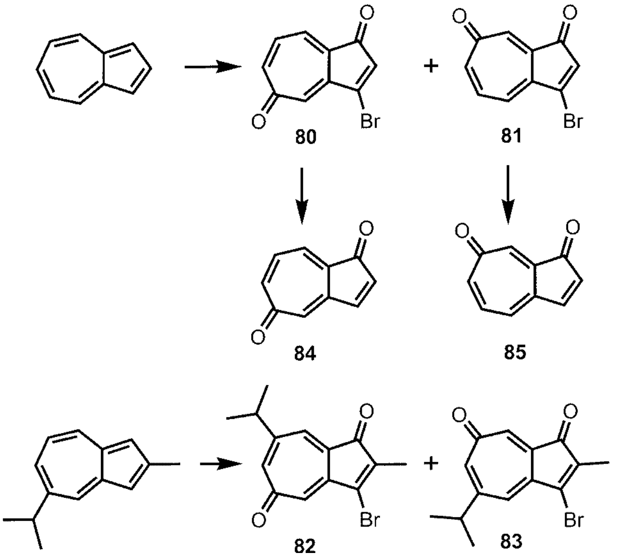


Scheme 25. Some oxidation products of guaiazulene



Scheme 26. Formation of 2-diazoazulenequinones

Being very interested by these observations, Nozoe set off on an investigation into the oxidation of azulenes. He found that both bromine and *N*-bromosuccinimide oxidized solutions of azulenes in aqueous THF to give two brominated quinones **80** and **81**, in 50−60 % yield after optimization[360,392] (Scheme 27). Molecular orbital calculations, X-ray crystallographic analysis, and the redox potentials[384] of these quinones were also studied. The analogous quinones **82** and **83** were obtained from 5-isopropyl-2-methylazulene. All of these bromoquinones could be reduced to the corresponding non-brominated quinones (e.g. **84** and **85**).



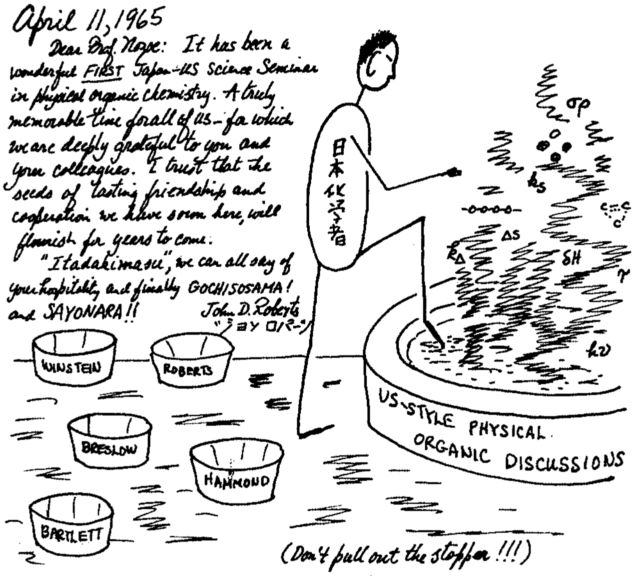
Scheme 27. The formation of azulenequinones

The results of these investigations were summarized in a 30-page article,[449] and published after Nozoe's death. It was an unforgettably moving scene for the authors to see him lying on his hospital bed and checking the galley proof of the paper.

V. Domestic and International Activities for the Stimulation and Dissemination of Organic Chemistry

Although Nozoe made some contributions as a member of the organizing committee for the 4th IUPAC Symposium on the Chemistry of Natural Products held in Kyoto in 1964, perhaps his greater contribution on that occasion was the organization of a two-day post-symposium meeting called the Sendai Colloquium. There were three parallel sessions of 15 invited lectures in Sendai, after which the attendees retired to a Japanese inn in Sakunami. After bathing in the nearby hot spring, a Japanese-style dinner was provided. Of course, everybody had to wear pure Japanese kimono and sit on a tatami floor. The after-dinner cultural exchange with a number of famous foreign organic chemists, was organized by Professor Koji Nakanishi, and greatly stimulated and activated the young Japanese chemists present, because previously they had had little chance to communicate with any foreign chemists.

In 1965, Nozoe invited and chaired the 1st Japan−U.S Cooperative Seminar on Physical Organic Chemistry in Kyoto. The American participants were Professors J. D. Roberts (the co-chairman), P. B. Bartlett, R. Breslow, G. S. Hammond, and S. Winstein; the Japanese counterparts were, besides Nozoe, Professors O. Simamura, T. Hayashi, Y. Yukawa, S. Oae, Y. Kitahara, and S. Itô. Associate professors of all the Japanese groups also attended. The atmosphere of the American-style discussion in the symposium was extremely well depicted by Professor Roberts' cartoon (Figure 3), which accompanied his comment to Nozoe. Although all the young Japanese participants had studied abroad for some time, they all were shocked by the vigorous discussion amongst the US participants.



April 11, 1965

Dear Prof. Nozoe: It has been a wonderful FIRST Japan-US Science Seminar in physical organic chemistry. A truly memorable time for all of us - for which we are deeply grateful to you and your colleagues. I trust that the seeds of lasting friendship and cooperation we have sown here, will flourish for years to come.

"Itadakimasu", we can all say of your hospitality and finally GOCHISOSAMA! and SAYONARA!!

John D. Roberts

Figure 3. Professor J. D. Roberts' comment on the 1st Japan−US Cooperative Seminar on Physical Organic Chemistry, April 1965

In 1966, soon after his retirement from Tohoku University, Nozoe accepted the job as the Far Eastern Regional Editor of *Tetrahedron* and *Tetrahedron Letters*, and became involved in the evaluation and final judgement of the manuscripts. It was also in this year that Nozoe and Professor M. Nakagawa started the Symposium on Nonbenzenoid Aromatic Chemistry, which was administered by the Chemical Society of Japan. The 1st annual meeting took place in Sendai, and was held in conjunction with the Symposium on Structural Organic Chemistry from 1969 up until 1987, at which point the two symposia were amalgamated, while keeping the name of the latter. This symposium has been held annually ever since.

In the early 1960s, the development of the chemistry of unsaturated seven-membered rings triggered the recognition of the importance of Hückel's rule, which was proposed in 1931 by Professor E. Hückel, and had induced the rapid evolution of synthetic (as well as theoretical) research on aromaticity. This had resulted in the synthesis of ring systems — from 3- to 18-membered and larger — in various places in the world. In 1961, Professor R. Breslow therefore proposed a new international symposium on nonbenzenoid aromatic chemistry in commemoration of Nozoe's retirement. This proposal was realized in August 1970, after some hesitation on the part of Nozoe. Thus, the International Symposium on the Chemistry of Nonbenzenoid Aromatic Compounds (ISNA), held in Sendai, was initiated by the Chemical Society of Japan under the auspices of IUPAC. Because Nozoe was the chairman of the organizing committee and Shô Itô was the general secretary, it had attracted virtually everybody interested in the new field.

Professor H. Zollinger, the IUPAC representative at this symposium, made the following report to IUPAC: "*This symposium was the best I have ever participated in all respects; organization, program, atmosphere, academic level, and administration. I would congratulate Professor Nozoe and his colleagues for the success of symposium.*" In his closing remarks, Professor Breslow made the following comment on behalf of the participants: "*... Professor Nozoe, in his opening remarks, kindly referred to the fact that in 1961, I suggested that a symposium on nonbenzenoid aromatic compounds be held in Japan. In his modesty, he did not tell you that I suggested that it be held* ***in his honor*** *on the occasion of his retirement. We all feel that this symposium has in fact been attributed to Professor Nozoe, even if this was never formally stated. His role in Japanese organic chemistry is revealed both in the world leadership of Japan in tropolone chemistry and more generally in the rapid progress of modern chemistry in Japan. I would like to ask the members of this symposium to express our appreciation with a standing ovation for Professor Nozoe. [Audience rose and applauded] The meeting in Sendai must be the first ISNA, and that a meeting so successful should be repeated...*"

His new proposal was accepted enthusiastically by the audience, and it was decided to hold the symposium every 3−4 years, the second one being scheduled for Germany. Despite the change in the name from "nonbenzenoid" to "novel" (thus keeping the same abbreviation), this symposium series has survived for more than 30 years; the 10th meeting was held in San Diego in 2001. Even though the topics discussed may change, we can be sure that this symposium will continue for a long time to come.

Nozoe had participated in every ISNA meeting until the 8th meeting in Braunschweig in 1995, and was the honorary member of the organizing committee in the 6th meeting in Osaka. He also made the following contributions; a set of plenary lectures entitled "Recent Advances in the Chemistry of Troponoids and Related Compounds in Japan"[432] at ISNA-1 and "Cyclohepta[*b*][1,4]benzoxazine and Related Compounds — Some Novel Aspects in Troponoid Chemistry"[443] at ISNA-4. However, what impressed the participants most was that he listened to all of the lectures from his seat in the front row of the lecture hall, and that he presented his newest results as a poster at every meeting himself, and enthusiastically discussed his chemistry with all those interested. Dr. P. Garratt, the editor of the collective volume of the ISNA-5 Plenary Lectures, wrote in his short preface "*Besides the distinguished plenary lectures published here, there was also an extensive and stimulating poster session, to which Professor Nozoe contributed, and well attending discussion...*"

After he passed away, Nozoe's friends and students decided to set up a named lectureship in his memory (at the five ISNA meetings to be held subsequently), supported by the Nozoe Memorial Trust. This movement must have originated from their affection and appreciation to him.

Apart from his tireless efforts to further international cooperation and the dissemination of chemistry, he was also inevitably involved in science administration. He served as a member of the Science Council of Japan for three years, but this post did not seem to fit him comfortably.

It should not be forgotten that he received young scholars and students from Taiwan and mainland China and financially supported many of them. This may have been one of his ways to express his appreciation to the people to whom he owed so much in his youth.

In 1961, Nozoe published a reference book in Japanese of about 700 pages[419−422] entitled "Nonbenzenoid Aromatic Compounds", as one of 25 volumes in the "Comprehensive Organic Chemistry" series. He also published a textbook (two volumes)[431,433] on organic chemistry in collaboration with some of his former students. Furthermore, following the progress of the chemistry of his field, he planned to publish, in English, a really comprehensive book on seven-membered aromatic compounds. Accordingly, he had collected more than 6000 references on these compounds and had drawn up a voluminous table of contents. Unfortunately the publication of this book was not realized in his lifetime. His son, Shigeo (also an emeritus professor of Tohoku University and a natural product chemist), said that Nozoe had expressed his regret on the matter during his hospitalization.

Although he enjoyed listening to classical music in his younger days, he did not have enough time to enjoy it later as much as he would have liked to. A non-smoker and non-drinker, his daily life was entirely devoted to nonbenzenoid aromatic chemistry. This point is pertinently expressed by Professor Hafner, who recalled his experience in Kyoto Prince Hotel, "*With amusement and admiration I still remember a phone call from the porter of the Hotel, which reached me one day in the very early morning, 'Professor Nozoe is waiting for you in the lobby.' An extended discussion during breakfast followed...*"[450]

However, his untold hobby was disclosed one day when Shigeo came to us with a notebook left by his father. It was Nozoe's diary written during his time as a visiting professor

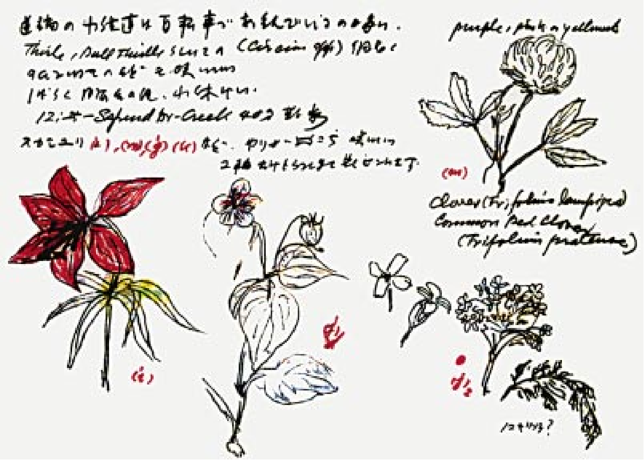


Figure 4. Nozoe's sketches of Canadian wild flowers (1971)

in Edmonton, Canada in the summer of 1971. With one glance, we were surprised not by his writing, but his sketches of Canadian wild flowers drawn on page after page. None of his students (including Shigeo) knew that he had a talent of this sort. Some of his sketches are shown in Figure 4. We believe that these sketches show Nozoe's individuality, and his affection for wild plants and to nature. He did not seem to have forgotten that his chemistry originated from the study of natural products, even after his long time period of research into the rather unnatural area of nonbenzenoid aromatic chemistry.

Finally, we would like to record two statements made by Nozoe. The first is an article published in Japanese in 1982 in *Chemistry and Chemical Industry*, the official journal of The Chemical Society of Japan, and is a message addressed to young Japanese chemists.

He wrote, "*Recalling my own experience over the past 50 years, I would like to address a few words to young Japanese chemists, especially graduate students in the field of organic chemistry. You are studying in excellent environment; to name but a few advantages, the substantial improvement of equipment in Japanese universities, the pertinent and kind advice of mentors, the ease of making contact with Japanese and foreign chemists and/or participating in domestic and international symposia and seminars. I have often been interested and pleased to read or listen to the presentation of their results on many occasions. I am also pleased by the favorable evaluations by foreign scholars and good reputations of young Japanese chemists. At the same time, however, I cannot help feeling that they might have omitted the unexpected phenomenon observed only once during repeated experiments in their research work, or that they might have ignored the experiments which they considered 'failed', in order to get a clean result, which can be obtained easily with modern equipment. On the other hand, there might be those who have difficult time breaking through in their research. I have known a rather large number of instances when, after many failures, the light broke through to sweep away the difficulty, or when an experiment which I overlooked led me in the right direction long afterwards. The history of chemical research also carries many, many instances of this kind. It is often said that, the more that things become convenient and useful, the busier you become and the less you think. It is a special pleasure and privilege given to a scientist interested in chemistry to be able to examine Mother Nature with curiosity and never-ending enthusiasm, to identify all products (not only the desired main product), and to follow reaction pathways by all applicable methods. You should always be careful not to overlook the slight indications that Mother Nature shows. A short detour is worth your time. I would like to conclude this short comment with an expression of great hope to the young people who are responsible for the progress of chemistry in the future.*"

The following words were spoken to his students from time to time, "*Research makes progress by thorough investigation. When stumble across a compound whose structure is hard to elucidate, or find a phenomenon which is unbelievable from your knowledge or common sense, it is the time to be pleased and excited, because it is telling you where something new lies, and it will lead you to a glorious discovery, after some struggle of course.*"

These words must be based on his own experience of single-handed research in his early days at Taihoku Imperial University. His genuine curiosity, directed to the uncovering of truth and enthusiasm for perfection, are clear from his many published articles.

Dr. J. I. Seeman, in his editorial note for Nozoe's autobiography, entitled "Seventy Years in Organic Chemistry",[447] stated, "*Tetsuo Nozoe is a man of boundless energy and great mental alertness — amazingly so for a man who was honored last year for attaining his Beiju (88th birthday)... Nozoe is a model of integrity, hard and dedicated work, inspiration, loyalty, and devotion... I wish my children could experience Tetsuo Nozoe as I have. He is a model for all of us.*"

In 1999, Nozoe's friends and students erected a heptagonal column (Figure 5) near the Chemistry Department at Tohoku University in order to symbolize the research activity on troponoid compounds that owes so much to the Nozoe's research group. By this gesture, we hope that many young people who study organic chemistry in the future will remember him and his activities.

Figure 5. Monument to troponoid chemistry

Finally, the authors can only express their sincere gratitude to Professor Nozoe for his wordless lessons given to us all the time by his attitude to chemistry, to nature and to people.

Thank you and goodbye, Professor Nozoe!

Toyonobu Asao (Sendai)
Shô Itô (Victoria)
Ichiro Murata (Nishinomiya)

Appendix

Honors Received by Professor Tetsuo Nozoe

1940: Sixth Order of Sacred Treasure (Government of Jappan)
1944: Majima Award (Chemical Society of Japan)
1944: Fifth Order of Sacred Treasure (Government of Japan)
1945: Fourth Order of Sacred Treasure (Government of Japan)
1952: Asahi Cultural Award (Asahi Press)
1953: Academy Award (Japan Academy)
1958: Order of Cultural Merit (Government of Japan)
1958: Man of Cultural Merit (Government of Japan)
1959: Honorary Citizen (Sendai City)
1962: Honorary Member (Chinese Chemical Society, Republic of China)
1967: Dark-blue Ribbon Medal and Imperial Cup (Government of Japan)
1972: Foreign Member, Royal Academy of Science, Sweden
1972: First Order of Sacred Treasure (Government of Japan)
1973: Honorary Member (Pharmaceutical Society of Japan)
1977: Honorary Member (Chemical Society of Japan)
1977: Honorary Member (Japan Society for Agricultural Chemistry)
1977: Honorary Member (Swiss Chemical Society)
1977: Member, Japan Academy
1979: Cultural Medal (Republic of China)
1981: August Wilhelm von Hofmann Memorial Medal (German Chemical Society)
1983: Honorary Citizen (City of Taipei, Republic of China)
1984: Special Award (The Society of Synthetic Organic Chemistry, Japan)
1990: Honorary Member (The Society of Synthetic Organic Chemistry, Japan)

List of Publications

A. Original Papers

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