FINSKA KEMISTSAMFUNDETS MEDDELANDEN

SUOMEN KEMISTISEURAN TIEDONANTOJA

REDAKTÖR – TOIMITTAJA
Gösta Brunow

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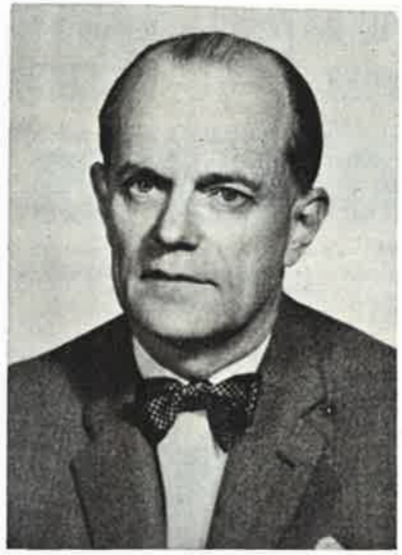

Anders Ringbom

in memoriam

Finska Kemistsamfundets och Suomalaisten Kemistien Seuras hedersledamot, professor emeritus Anders Ringbom, avled i Åbo den 21 december 1972.

Anders Ringbom föddes i Åbo 1903. Efter avslutad skolgång började han sina studier vid Åbo Akademi och avlade diplomingenjörsexamen år 1925. Han disputerade för teknologie doktorsgraden vid Åbo Akademi år 1936.

När han den 1 januari 1969 avgick som emeritus hade han i 42 års tid varit verksam vid Åbo Akademi, först som forsknings- och undervisningsassistent och senare som lektor och e.o. professor i analytisk och oorganisk kemi. Till ordinarie innehavare av professuren i kemi, företrädesvis analytisk och oorganisk kemi, kallades han år 1952.

Hans vetenskapliga produktion vittnar om gedigen kunskap och eminent vetenskaplig förmåga. Hans artiklar präglades av klarhet i framställningssättet och han hade ett utpräglat sinne för att finna eleganta lösningar till komplicerade analytiskkemiska problem. Han ställde höga krav på innehåller i sina skrifter och baserade framställningen på teoretiska beräkningar. Det är därför inte förvånande att de rönte stor uppskattning bland analytiska kemister. Flera av hans arbeten är i dag klassiska. Sin största insats gjorde Anders Ringbom vid utvecklandet av den teoretiska bakgrunden för komplexometrisk analys, dvs. bestämning av metalljoner i lösning. Sedan början av 1950-talet intresserade han sig för denna analysmetod och visade hur man på ett enkelt och elegant sätt kunde bemästra de matematiska problemen. Han presenterade sin metod i form av en artikel och senare som ett kapitel i ett större kompilerande verk. Vartdera arbetet rönte stor uppskattning och Anders Ringbom inbjöds till flera länder för att föreläsa om den nya tekniken.

I monografin »Complexation in Analytical Chemistry», som utkom år 1963, utvidgade han metoden att gälla för olika typer av kemisk analys. Boken påverkade i hög grad den teoretiska analytiska kemin och som ett tecken på dess uppskattning kan nämnas att den översatts både till japanska och till franska. År 1966 tilldelades han av Finska Vetenskapssocieteten professor E. J. Nyströms pris som en erkänsla för gediget vetenskapligt arbete. Samma år erhöll han även av Åbo Akademis konsistorium lärarstipendiet ur Harry Elvings legat. Tre år senare utsågs han att i Stockholm hålla en s.k. Torbern Bergman-föreläsning och erhöll vid detta tillfälle Torbern Bergmanmedaljongen för eminent vetenskaplig verksamhet.

Efter sin pensionering var han aktivt verksam och intresserade sig speciellt för bestämning av jämviktskonstanter för olika komplexbildningsreaktioner. Han höll även på med att utarbeta en ny upplaga av sin uppskattade monografi.

Hans analytisk-kemiska kunnande har även utnyttjats i internationella sammanhang. Som medlem av den analytiska sektionen i »International Union of Pure and Applied Chemistry» har han aktivt deltagit i utarbetandet av tabeller rörande olika metallsulfiders löslighet.

Anders Ringbom var icke enbart verksam inom sitt fackområde. Vid sidan av förtroendeuppdrag inom Åbo Akademi, han var dess prorektor 1954 – 57 och dekanus för kemisk-tekniska fakulteten 1962 – 66, har hans vidsynthet och klara omdöme utnyttjats även av statsmakten. Han var medlem av Statens teknologiska kommission åren 1961 – 65 och utsågs av undervisningsministeriet till sakkunnig i frågor rörande den tekniska undervisningen åren 1965 – 67. Han intresserade sig även för studentlivet och var president för Åbo Akademis Studentkår åren 1953 – 57 och valdes till hedersledamot år 1957.

Anders Ringbom var högt värderad som vän och kollega. Med sitt angenäma och gemytliga väsen spred han en atmosfär av trevnad kring sig. Hans minne skall länge leva bland dem som hade förmånen att räknas bland hans vänner.

Erkki Wänninen

Ragnar Lydén

in memoriam

Professor, hedersdoktor vid Medicinska fakulteten Ragnar Lydén avled den 13 december 1972 i en ålder av 80 år. Med honom gick ur tiden ett av våra få kvarblivna universitetsoriginal av klassisk typ. Han gjorde Helsingfors universitet stora tjänster som assistent, docent, biträdande professor och chef för kvalitativa avdelningen vid kemiska institutet under fyrtiotre år och i synnerhet som briljant föreläsare i tre fakulteter och dessutom vid farmaceutiska institutet och gymnastikinrättningen under mer än hundra terminer eller över femtio år. Antalet av hans elever måste ha rört sig mellan 20 000 och 30 000 eller inemot en procent av Finlands befolkning. Detta skedde under en period då lärarbristen var katastrofal och universitet och stat hänsynslöst vräkte en förkrossande börda av massundervisning på äldre assistenter och biträdande professorer, som till hjälp hade blott oerfarna, tillfälliga assistenter, med en lön som per år i realvärde uppgick till ungefär det belopp som en undervisningsassistent nu bär upp per månad. Lydén förmådde bära denna börda med hjälp av sitt temperament och sin oförbrännerliga humor, som var både elever och vänner till stor glädje.

Han var född i Vampula som son till kronofogden K. R. Lydén. Vampula gränsar i norr till Vittis (Huittinen); varifrån Risto Ryti var hemma. I Helsingfors kom de båda herrarna att arbeta nära varandra vid Snellmansgatan, Ryti i Finlands bank och Lydén på Kemikum. Lydén berättade gärna om sina sammanträffanden och diskussioner med den blivande presidenten.

Lydén blev student från Svenska reallyceum i Åbo 1911, och fil.mag. vid Helsingfors universitet vårvintern 1917. Då hade han redan över ett år tjänstgjort som extra assistent vid universitetets kemiska institut. Han var sedan assistent vid Kemikum ända till 1930, blev filosofiedoktor 1926, docent 1928 och biträdande professor (adjunkt) i kemi 1930–1959. År 1920 var han kemistassistent vid Helsingfors vattenverk, från samma år lärare i kemi vid gymnastikinrättningen och 1918–1921 assistent också vid agrikultur-forstvetenskapliga fakultetens propedeutisk-kemiska laboratorium. Han var kurator för Åbo nation 1920–1921 och kemilärare vid Nya svenska läroverket i Helsingfors 1924–1928. Notarie i matematisk-naturvetenskapliga sektionen var han under åtskilliga år från 1925. På uppdrag av Finlands apotekarförening föreläste han kemi och farmaceutisk kemi för farmacieelever 1928–1938 och för medikofil- och medicinestuderande på förordnande ända till 1965 som pensionerad, tidtals utan ersättning för att inte få så höga skatter!

Han var ledamot av Soc. pro fauna et flora fennica och sedan tidigt datum också av Finska kemistsamfundet, ehuru hans sprakande temperament fick honom att skriva ut sig ur samfundet emellanåt. Han kunde ha utpräglade sympatier och antipatier och avge drastiska omdömen om personer, t.ex. »inkompetensens inkarnation».

Hans första egentliga publikationer (1921–1923) gällde oxalsyrans fotolys i närvaro av uranylsalter. Doktorsavhandlingen 1925 gällde etrars spjälkning med acylbromider, ett tema som han under namnet undersökningar över kolväteradikalers syreaffinitet fortsatte i ytterligare tre avhandlingar. Senare arbetade han med talliumjodider, krom-3-oxid, bl.a. med dess oxidation och upplösning under inverkan av silveroxid, och med olika uranföreningar. Hans sista publikation är från 1965.

Lydéns bästa arbeten var hans oorganiska där han studerade flere kuriösa och intresanta reaktioner och var en skicklig och uppfinningsrik forskare i klassisk stil, som ej hade större intresse för fysikalisk-kemiska tolkningar. Detta märktes också i hans organisk-kemiska arbeten, där skillnaderna mellan reaktionshastighet och affinitet inte alltid var så klar. Detta har på sätt och vis samband med andan i det svar han vid en studenttillställning gav en medikofilare, som var så berusad att han inte kände igen Lydén utan skrek »vad grymtar du där, du din gulnäbb». Vartill Lydén röt »jag är filosofiedoktor och behöver aldrig

mera studera!» – Till Lydéns försvar kan anföras att han dock stödde sig på uppfattningar publicerade bl.a. av J. v. Braun ännu 1922. Ett elakt rykte förtäljer bl.a. att Lydén som gammal uranforskare år 1945 blankt vägrade tro på att atombomben skulle bygga på sönderdelning av uran. Senare fick han på grund av atombombsfaran själv lov att begära licens för inköp av uransalter för sina analytiska arbeten. Han måtte ha ansetts ofarlig, ty han fick licenserna utan svårighet.

I samband med sin undervisning gav han ut läroböcker i kemisk analys och stökiometri.

Hans sinne för humor gjorde honom till stor diktare och kringspridare av anekdoter i burlesk stil, ofta i många verser och avsjungna med stor bravur. Under kriget uppfångade eller gjorde han otaliga historier riktade mot nazisterna, av vilka bl.a. den kände estetikprofessorn Yrjö Hirn ofta lär ha njutit. Lydéns mimiska framställning av apornas minspel på Högholmen fick många av hans vänner att skratta sig halft fördärvade. Liknande härmningsnummer av även högt ställda akademiska medborgare hörde också till hans repertoar. Hans studentikosa läggning och trycket av massundervisningen gjorde honom till ett slags föregångsman för kollektiv undervisning. Mer än en gång gick hans tentamina för grupper av medikofilare så till att han gav en fråga till en tentand. Var svaret rätt hette det »nå, gruppen tycks kunna, alla är godkända». I motsatt fall hette det »gruppen tycks inte kunna någonting, kom tillbaka nästa vecka.»

Att Lydén dock hade ambitioner att hålla undervisningen på hög nivå framgår av att han var ytterst förgrymmad när institutets prefekt en gång på 1930-talet, irriterad över klagomålen över lång studietid på kvalitativa avdelningen, gav order om att påteckning skulle ges för olika vitsord helt enkelt på basen av de tider studenterna tillbragt på avdelningen!

Lydéns »demoniska» Döbeln-utseende med det svarta håret och polisongerna och de djupt liggande brinnande ögonen utövade i förening med hans evigt unga temperament en romantisk attraktion på åtskilliga unga damer, vilket också ledde till åtskilliga giftermål.

Lydén var en intresserad och skicklig blomsterodlare och hans stenpartier på hans parcell i Domsby var en sevärdhet.

Ragnar Lydéns vänner och många tusen elever minns honom som den temperamentsfulle, briljanta föreläsaren och evigt unga, livliga och egenartade människan.

Terje Enkvist.

Distribution and Composition of Extractives in Wood.

7. An Improved Method for the Determination of Unsaponifiable Components in Pine *(Pinus silvestris)*.

Dan Manell and Göran Pensar

The Institute of Wood Chemistry and Cellulose Technology, Åbo Akademi, Åbo, Finland.

Abstract

A method of isolating the unsaponifiable components from different zones in the trunk cross sections of Scandinavian Pine and the consequent determination of their radial distribution and composition is described. Quantitative gas chromatography was carried out using the "internal standard method" and the relative weight responses (RWR) for the main components were established. β-Sitosterol was quite dominating in each investigated fraction. In the outer sapwood it constituted 82.3 % of the weighed unsaponifiables, in the sapwood 77.7 %, in the border zone 61.0 % and in the heartwood 39.9 %. The corresponding values for pinosylvin monomethyl ether were 2.3 %, 2.0 %, 3.6 % and 16.1 %. The identification of resin acid methyl esters, cycloartenol, methylene cycloartanol and α-sitosterol was made with a LKB 9000 gas chromatograph – mass spectrometer. The presence of these compounds in pine wood extractives have not to our knowledge been reported in previous investigations.

Introduction

The radial distribution and composition of the lipophilic part of the extractives (e.g. the wood resin) in common Scandinavian softwoods used as raw material in pulp manufacture (*Pinus silvestris* and *Picea abies*) has previously been studied by Pensar[1]. In his investigation compounds classified as unsaponifiables were only determined as a group, without any effort to study the composition of this fraction. The aim of this study is to continue that investigation by outlining the general trends in the radial distribution and composition of the dominating unsaponifiable compounds in resin from pine wood. According to the classical procedure of separation, the unsaponifiables can be defined as the compounds separated after alkaline hydrolysis (or "saponification") as a fraction insoluble in water, but soluble in nonpolar organic solvents. The main components of this fraction from wood resin are: hydrocarbons, aliphatic and cyclic alcohols and carbonyl compounds, but also phenolic substances having a significant solubility in these solvents may be found in the fraction.

The unsaponifiables are very complex in their composition and it was therefore necessary for this study to develope an analytical method by which a great number of samples could be treated without time consuming and laborious work, but with proper accuracy in the determinations.

Experimental

Chemicals. The column chromatographic separations were performed using silica gel of size less than 200 mesh (E. Merck AG). Qualitative thin-layer chromatography was carried out using commercially available 20 × 20 cm plates precoated with 0.25 mm layers of silica, with or without impregnation of fluorescence indicator (E. Merck AG). The gas chromatographic analyses were made using silanized glass columns filled with 1 % SE 30 on acid washed and silanized Chromosorb W (80/100 mesh) as support. Trimethylsilyl ether (TMS) derivatives of the unsaponifiables were made by dissolving them in tetrahydrofuran and using bis(trimethylsilyl)trifluoracetamide (BSTFA, Fluka AG) as silylating agent. Reagents and solvents used were of analytical purity and the latter were percolated through alumina and redistilled before use. For identification purposes and for the determination of the gas chromatographic relative weight responses the compounds listed in table 1 were used.

Table 1. Reference compounds used in this study.

Compound	Source
Squalane	E. Merck AG
Dehydroabietal	Synthetized at the institute
Dehydroabietol	»
Cholesterol	Fluka AG
β– Sitosterol	Applied Science Lab., Inc.
Fatty alcohols (C14, C16, C18, C20, C22, C24)	Applied Science Lab., Inc.
Hydrocarbons (C24, C25, C26, C28)	Fluka AG
Stilbestrol	Koch Light Lab.Ltd.

Material. In order to check the suitability of the method on native material one 85 year old pine originating from the southern part of Finland (Tuusula, Ruotsinkylä), was cut in January 1972. Four cross sections were taken at the level of four meter in the trunk. After examinating the discs under ultraviolet light the border between sap- and heartwood was established whereafter the wooden parts of the discs were separated into following concentric zones: young sapwood (O) 5–10 annual rings, sapwood (M), border zone (C–M) 5–10 annual rings located closest to the heartwood and heartwood (C). The wood samples were after drying immediately ground to 20 mesh and extracted.

Apparatus. The gas chromatographic analyses were carried out using a Varian Aerograph Autoprep 705 instrument equipped with flame ionization detector. In order to get a sufficient degree of reproducibility with this instrument following modifications were made. To prevent thermal degradation and band-spreading of the sample a 3.0 m glass column with an i.d. of 1.8 mm allowing "on-column" injection was installed, and furthermore the connection tube to the detector was filled up with column packing material. A programming unit for the oven temperature was also installed.

The quantitative measurements were made with a Disc-integrator fitted with an automatic printer and the sensitivity and reproducibility of the GC system were continually tested with a solution of 1 % alcohol in carbon disulfide.

In connection with the preparative thin-layer separations a Camag Chromatocharger was used for sample application.

The mass spectrometric identifications were made with a gas chromatograph-mass spectrometer, model LKB 9000.

The analytical method. A flow scheme illustrating the analyses of the unsaponifiables is shown in figure 1. For each analysis about 20 g wood meal were extracted in a Soxhlet apparatus with benzene for 3.5 h. The solvent was carefully removed by distillation at reduced pressure under nitrogen, not allowing the temperature to exceed 50°C. The extractives were weighed and the extractive content calculated as percent of dry wood, whereafter the extractives were immediately dissolved in petroleum ether (b.p. 30–40 °C)-(10 ml petroleum ether/100 mg extractives), the solution was filtrated and the filtrate was taken to dryness. The petroleum ether solubles were determined as above.

The extractives were saponified for 3.5 h at 74.5 °C in 20 ml 0.44 M ethanolic (94 %) potassium hydroxide. The cooled solution was neutralized with 1.28 M ethanolic HCl and filtrated, the salt was washed with ether whereafter the solution was taken to dryness and redissolved in 3 ml of ether. This sample was applicated on a column (2 × 21 cm) containing alkaline silica gel, prepared according to Paasonen[2]. The unsaponifiables were eluted with 200 ml diethyl ether and collected in the eluate, thus separated from the acids which remained in the column. The amount of isolated unsaponifiables was determined as stated above for the extractives.

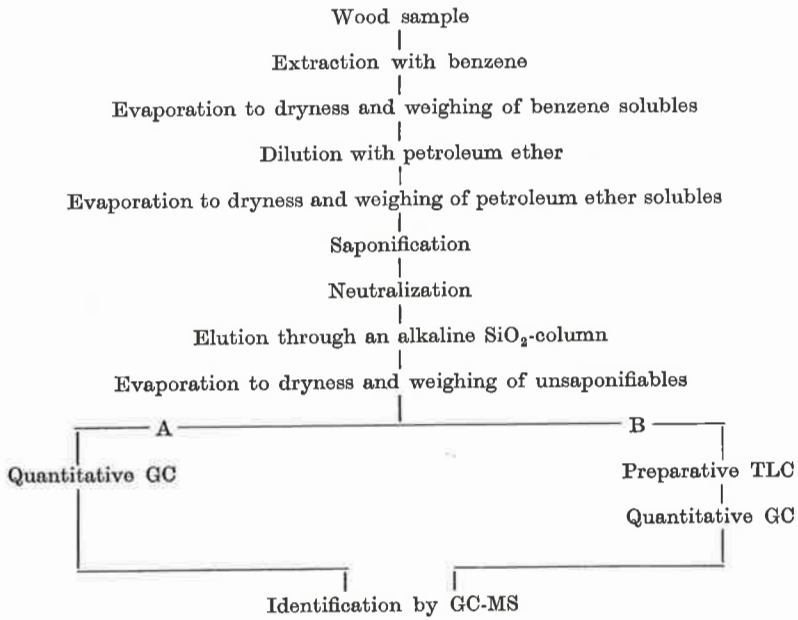

Figure 1. A flow scheme illustrating the analytical method. The route "B" was used for the identification of some minor components and was not subjected to all samples.

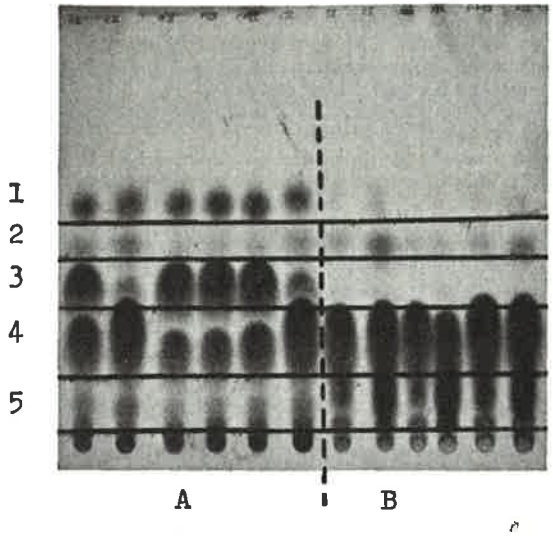

Figure 2. TLC-plate showing the course of the saponification. The eluting solvent mixture was petroleum ether — diethyl ether (75 : 25, vol : vol) and the main spots represent 1: waxes and hydrocarbons, 2: diterpene aldehydes, 3: esters of triglyceride-type, 4: free acids, 5: sterols, fatty- and diterpene alcohols. The samples applicated are A: extracts before and B: after saponification.

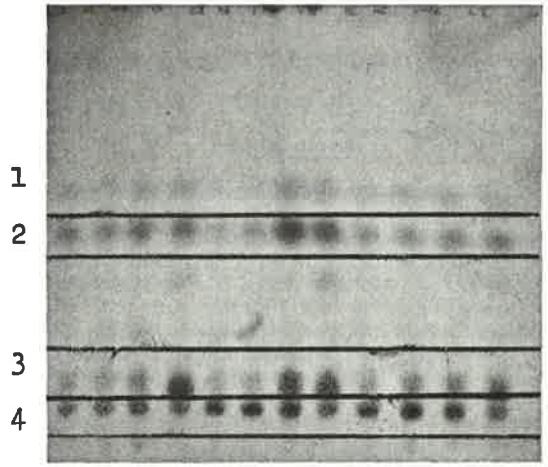

Figure 3. TLC-plate showing the unsaponifiables after the column separation. The main spots represent 1: hydrocarbons, 2: diterpene aldehydes, 3: fatty- and terpene alcohols, 4: sterols.

The course of the saponification and the column chromatographic separations were controlled by TLC checks as shown in figures 2 and 3.

The unsaponifiable fraction of the extractives was hereafter analyzed by GC. However some overlappings with the minor components were unavoidable as can be seen from the gas chromatograms in figure 5. Thus a more adequate picture of these could be achieved if the fraction was furthermore separated into subfractions by the aid of preparative TLC. The sample, dissolved in benzene, was applicated as a narrow stripe onto a plate impregnated with a fluorescense indicator and developed in two steps. In the first step a nonpolar solvent mixture was used (petroleum ether: ether in 95 : 5 vol/vol) and the solvent front was allowed to rise to the upper line of the plate. With this system hydrocarbons, aldehydes and compounds with Rf-values close to these were separated from the rest of the material. The stripes were visualized under ultraviolet light (254 nm) and removed from the plate for GC analyses. The plate was then eluted in a second step with a more polar solvent mixture (petroleum ether : ether in 50 : 50 vol/vol) and the solvent front was allowed to rise half of the plate separating fatty and diterpene alcohols from sterols and leaving highly polar material at the starting point. The stripes were then treated as above. In the GC analyses each sample was run both in free form and as TMS-derivative, which was prepared using BSTFA as silylating agent. Complete silylation was achieved after keeping the sample at 60°C for 30 min. In the GC analyses the injector and the detector temperatures were kept at 290°C and the temperature program used in the separations is shown in figure 4. Nitrogen (25 ml/min) was used as carrier gas.

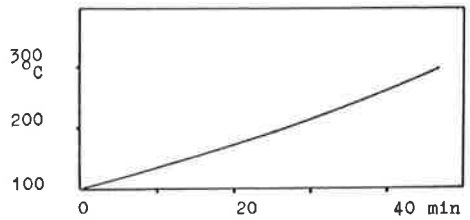

Figure 4. The temperature program used for the GC-separations.

Results and discussion

The separation obtained with the gas chromatographic system is shown in figure 5 and in table 2 are listed the compounds which have been identified in the unsaponifiable fraction and their amounts in the different parts of the trunk cross section.

The identity of the hydrocarbons (GC-peaks 1, 13, 14 and 18), aldehydes (GC-peaks 2 and 3) and alcohols (GC-peaks 5, 6, 10, 15 and 17) were determined by comparing their GC retention times and mass spectra with those of syntethized reference compounds[3]. Methyl dehydroabietate and methyl abietate (GC-peaks 9 and 11) were similarily identified by comparison with mass spectra found in the literature[4].

When the samples were run in free form, the mass spectrum of GC-peak no. 12 had the most abundant peaks in the high mass

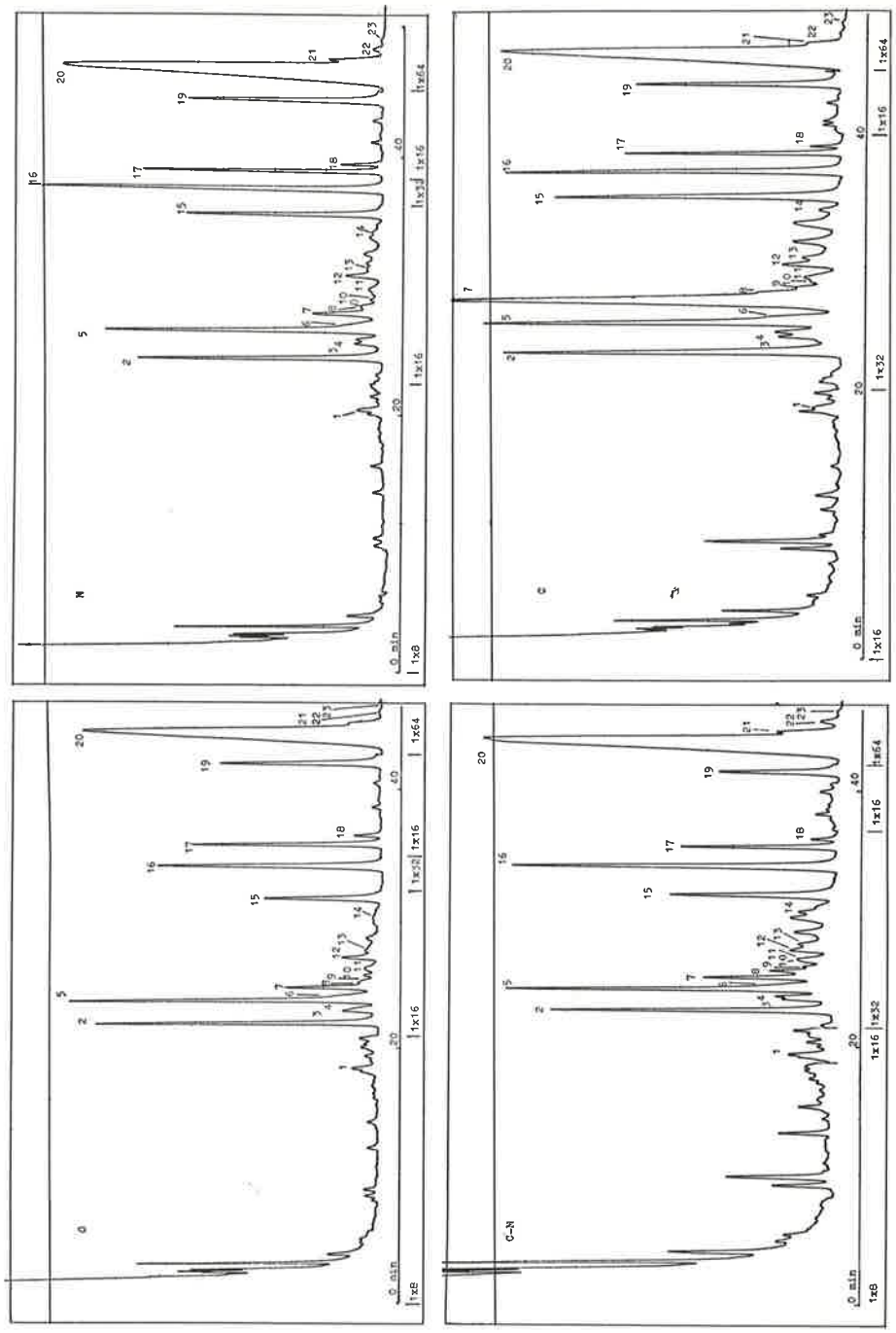

Figure 5. Gas chromatograms illustrating the composition of the unsaponifiables in different zones of the trunk cross sections. O = outer sapwood, M = sapwood, C-M = border zone between sapwood and heartwood, C = heartwood. For the identity of the peaks, see table 2.

range at m/e 312 (molecular ion), 297 (M-15), 253 (M-59) and 237 (M-(15 + 60)). Compared with the corresponding peaks in the mass spectrum of methyl dehydroabietate[5] it was noticed that the peaks for the unidentified compound showed a decrease of two mass units, thus indicating a fourth double bond. Except

Table 2. Composition of unsaponifiable fraction in different zones of the trunk cross sections. The amount of a compound is expressed as percent of weighed unsaponifiables. The samples were chromatographed as TMS-derivatives.

Peak no.	Compound	O %	M %	C-M %	C %
1	Pimaradiene	+[a]	+	+	+
2	Pimaral	4.6	3.5	5.6	6.2
3	Isopimaral	0.9	0.7	1.6	1.6
4	Pinosylvin dimethyl ether	0.4	0.8	1.4	1.5
5	Pimarol	4.0	3.0	4.9	4.8
6	Isopimarol[b]	0.9	0.7	1.2	1.1
7	Pinosylvin monomethylether	2.3	2.0	3.6	16.1
8	Dehydroabietol	0.7	0.7	1.2	0.7
9	Methyl dehydroabietate[c]			+	0.5
10	Abietol[d]	+	+	1.6	1.2
11	Methyl abietate	0.5			
12	Dehydrodehydroabietol / Methyl dehydrodehydroabietate	0.8	0.6	1.5	1.1
13	Tetracosane	+	+	1.2	1.1
14	Pentacosane	+	+	0.9	1.5
15	Lignoceric alcohol	1.8	2.3	3.8	4.2
16	Squalane	Internal standard			
17	Behenic alcohol	2.5	2.5	2.4	1.9
18	Squalene[e]	0.3	0.6	0.5	0.5
19	Campesterol	3.2	3.1	2.7	1.6
20	β-Sitosterol	82.7	77.7	61.0	39.9
21	Cycloartenol	1.8	2.3	2.0	0.7
22	Methylene cycloartanol	+	0.7	0.65	+
23	α-Sitosterol	+	+	+	+

a. + = less than 0.5 %. *b.* Due to overlapping, isopimarol was preferably determined when the sample was run in free form. *c.* The presence of methyl pimarate and methyl isopimarate were indicated when mass spectra from an aldehyde fraction separated by TLC (route B in fig. 2) were taken. One should note that methyl esters are no true unsaponifiable constituents of the extract but are counted as they go along in the analytical procedure. *d.* MS showed that small amounts of tricosane was present in this peak. *e.* MS showed small amounts of octacosane in this peak.

for the differences of two mass units the general appearance of the two spectra was the same and the unknown compound could be methyl dehydrodehydroabietate. When the samples were run as TMS- derivatives, the GC-peak no. 12 was overlapped by a compound which seemed to be the corresponding alcohol to methyl dehydrodehydroabietate. This alcohol had the most abundant peaks in the high mass range at m/e 356 (molecular ion), 341 (M-15) and 251 (M-(15+90)). In the central part of the spectrum there were three dominating peaks at m/e 183, 171 and 157.

The mass spectra of a dimethoxy stilbene, probably pinosylvin dimethyl ether, cycloartenol, methylene cycloartanol and α-sitosterol (GC-peaks 4, 21, 22 and 23) were in good accordance with those earlier recorded at the institute in connection with an investigation on tall oil[6]. Pinosylvin monomethyl ether (GC-peak 7) was run in free form and as TMS-derivative and its mass spectra showed the expected base peaks at m/e 226 and 298 (molecular ion) respectively. It was identified by comparison with the corresponding dimethyl ether. According to preliminary MS studies the two peaks which are abundant in heartwood, having retention times about 10 min should be α- and δ-murolene. The less abundant peaks between squalene and campesterol are unidentified aliphatic hydrocarbons.

To achieve a realistic quantitative determination of the unsaponifiable constituents from the GC analyses it is necessary to establish their relative detector responses. As pointed out by Onkiehong[7] the response of the hydrogen flame ionization detector is proportional to the weight percentage of carbon in organic substances and the sensitivity of the detector decreases as soon as atoms other than carbon and hydrogen are introduced into the molecule. This decrease cannot be regarded as constant for each heteroatom. For this reason the detector should be calibrated for every component injected in the gas chromatograph.

The formulas 1a. and 1b. give the relative weight response (RWR) of a component against an internal standard and the concentration of the component.

1a. $$RWR_u = \frac{C_s}{C_u} \times \frac{A_u}{A_s}$$

1b. $$C_u = \frac{C_s}{RWR_u} \times \frac{A_u}{A_s}$$

Here A_u and A_s denotes for the areas, C_u and C_s for the concentrations of the unknown and the internal standard respectively.

In table 3 is given the RWR values that has been determined in this investigation using squalane as internal standard. The GC conditions during the response determinations were the

same as when the unsaponifiables were analyzed. The technique used in these determinations was the same as that described earlier in the investigation of RWR values of chlorinated fatty acids[8]. The relative standard deviation was here found to be less than 1.6 %.

Table 3. Response values for reference compounds in free form and as TMS-derivative.

Compound	Mean RWR Free form	Mean RWR TMS-derivative
Squalane (internal standard)	1.00	–
Dehydroabietal	0.80	–
Dehydroabietol	0.86	0.99
Cholesterol	0.88	0.99
β-Sitosterol	0.52	0.65
Octadecanol	0.90	1.10
Stilbestrol	0.66	–

In the calculations of the composition of the unsaponifiable components which are given in table 2, these RWR values were used. Following approximations were made: the values for dehydroabietal and dehydroabietol were used for all diterpene aldehydes and alcohols respectively. In the same way the value for octadecanol stood for all the fatty alcohols and that for stilbestrol stood for pinosylvin dimethyl and monomethyl ether. The RWR value for campesterol (as TMS-derivative) 0.85 was calculated from the values for cholesterol, β-sitosterol and in the literature[9] given value for campesterol.

Considering the components that form the main part of the unsaponifiable fraction, it can be seen from table 2 that in the sapwood β-sitosterol and in the heartwood β-sitosterol together with pinosylvin monomethyl ether are quite dominant. Their radial variation within the fraction is remarkable, as the former shows a decrease and the latter an increase in direction into the trunk. Similar trends can be found for campesterol (decrease), cycloartenol (decrease), pinosylvin dimethyl ether (increase) and lignoceric alcohol (increase).

It is worth noticing that compounds considered to be typical of the heartwood e.g. the pinosylvines are also distributed in the sapwood.

Resin acid methyl esters were also detected in the unsaponifiable fraction. Their presence in pine wood extractives have not to our knowledge been reported in previous investigations. This is also the case with cycloartenol, methylenecycloartanol and α-

sitosterol. Their presence could be predicted as they recently were found to occur in tall oil[6].

When minor components in the diterpene aldehyde, diterpene alcohol and hydrocarbon fractions were investigated using further subfractioning of the unsaponifiable fraction by preparative TLC (route B in figure 1), the presence of dehydroabietal, palustrol, neoabietol, tricosane, hexacosane and heptacosane could be confirmed by GC-MS.

Holmbom and Avela[6] have pointed out the important role the unsaponifiables play in tall oil refining, as they contribute to the unwanted pitch formation. Furthermore the diterpene aldehydes and pinosylvines may codistil with the tall oil fatty acids and remain as inpurities lowering the quality of the final product. Therefore informations gained from investigations clarifying the relation between the structure of wood and its resin content should contribute to the thorough knowledge of pine wood, used as raw material in the pulp industry.

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Literature

1. Pensar, G., "*Studies on the Radial Distribution and Composition of Wood Resin in Cross Sections of Scandinavian Spruce and Pine*". *Thesis*, Åbo 1969.
2. Paasonen, P., "*On the Non-Volatile Ethyl Ether Extractives of Birch Wood and the Changes in Composition Effected by Aging and Sulphate Pulping*". *Thesis*, Helsinki 1966.
3. Johansson, B., and Bruun, H., *Unpublished results.*
4. Zinkel, D. F., Zank, L., and Wesolowski, M. F., "*Diterpene resin acids*", U. S. Department of Agriculture, Forest Service, Forest Product Laboratory, Madison, Wis. 1971.
5. Enzell, C. R., and Wahlberg, J. *Acta Chem. Scand.* **23** (1969) 871.
6. Holmbom, B., and Avela, E. *Acta Acad. Aboensis* **B 31**, 16 (1971) 1.
7. Onkiehong, L., "*The hydrogen flame ionization detector*". *Thesis*, Amsterdam 1960.
8. Pensar, G., Manell, D., Ketola, M., and Pihlaja, K. *Acta Chem. Scand.* **25** (1971) 1489.
9. Rozanski, A., *Anal. Chem.* **38** (1966) 36.

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An Electron Resonance Study of the Anion Radical of 3,3′-dimethoxybiphenyl.

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Abstract:

The anion radical of 3,3′-dimethoxybiphenyl has been prepared by alkali metal reduction in 1,2-dimethoxyethane and tetrahydrofuran using high vacuum technique. It is shown that this anion radical is also formed under the influence of the alkali metal if 3,4,3′,4′-tetramethoxybiphenyl is used as parent compound through irreversible cleavage and replacement with hydrogen of the methoxy groups in the 4 and 4′ positions. The electron resonance spectrum of the anion radical has been recorded at different temperatures. The recorded spectra can be interpreted as the superimposed spectra of two different species, which are proposed to be the *cis* and *trans* isomers. The assignment of the coupling constants to specific positions in the molecule were done with the aid of INDO molecular orbital calculations.

Introduction

Previously the cation radical of 4,4′-dimethoxybiphenyl has been investigated and its electron resonance spectrum at lower temperatures interpreted assuming *cis-trans* isomerism [1,2]. With the methoxy substituents in the meta position with respect to the central C-C bond, no additional steric interactions are introduced. Any difference in the behaviour of the anion radical of 4,4′-dimethoxybiphenyl[3] and 3,3′-dimethoxybiphenyl with respect to the restricted rotation about the coannular bond should therefore be attributed to the difference in conjugation between the two aromatic rings (different torsional barrier), to the difference in the reduced moment of inertia of the top about the central C-C bond, and to the different degree of interaction with the solvent molecules due to the different shape of the molecules.

In the present paper an electron resonance study of the anion radical of 3,3′-dimethoxybiphenyl with particular emphasis on the hindered rotation is presented.

Experimental

The sample of 3,3′-dimethoxybiphenyl was a commercial Purum grade product from Fluka AG, chromatographically pure and therefore used as such.

The sample of 3,4,3′,4′-tetramethoxybiphenyl was prepared from 4-iodoveratrole through the usual Ullman reaction, according to Seer and Karl.[4] The product was recrystallized from methanol and finally sublimed repeatedly at 100°C and 0.04 mm Hg until the melting point remained constantly 130°, (lit.[4] 130 – 132°C). The purity of the sample was also checked by gas-liquid chromatography.

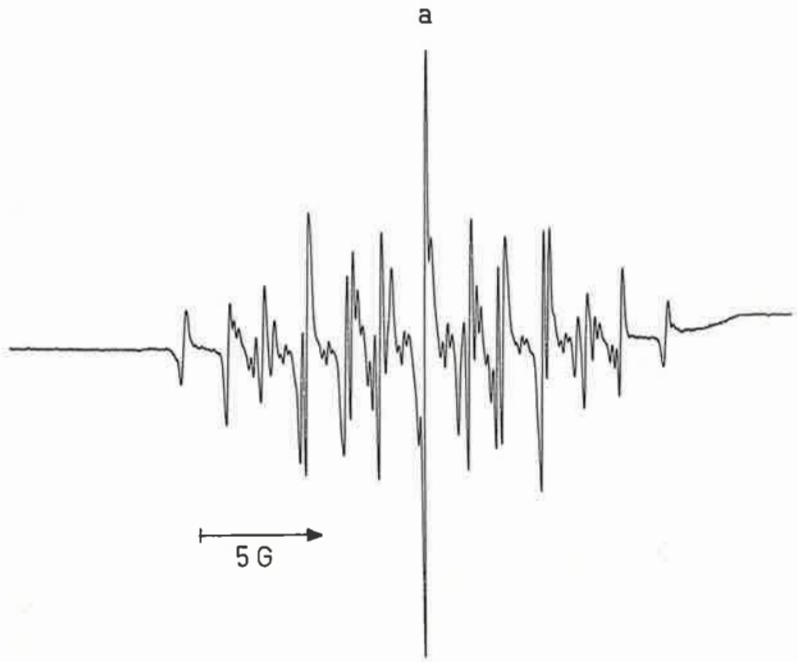
The anion radical was generated by chemical reduction of the parent compounds employing the high vacuum technique described previously [3,5] The recording conditions for the spectra, the INDO MO calculations, and the computer simulations of the one and two component electron resonance spectra are described elsewhere[2,3].

Results and Discussion

The anion radical of 3,3′-dimethoxybiphenyl can be prepared using two different parent compounds. If 3,3′-dimethoxybiphenyl is used, the reduction with alkali metal in both 1,2-dimethoxyethane and tetrahydrofuran proceeds as a one-electron reduction giving the desired anion radical. However, also 3,4,3′,4′-tetramethoxybiphenyl (diveratrole) can be used as parent compound. The reduction with alkali metal then proceeds presumably through several reaction steps where the net result is that the two methoxy groups in the 4 and 4′ positions are replaced with hydrogen atoms and the anion radical of 3,3′-dimethoxybiphenyl is formed. Such cleavage and replacement with hydrogen, or coupling of the fragments formed, has been observed by several authors when methoxysubstituted aromatic compounds are treated with alkali metals in aliphatic ether solvents [6–10]. Recently the formation of biphenyl from 4,4′-dimethoxybiphenyl (pp'-dianisole) under such conditions was observed in connection with the study of the anion radical of pp′-dianisole.[3] But whereas pp′-dianisole was resistent (with regard to the cleavage reaction) to sodium in a mixture of 1,2-dimethoxyethane and tetrahydrofuran (1 : 1 by volume), cleavage of the 4,4′ methoxy groups in diveratrole occurred also in this medium, indicating that the additional 3,3′ methoxy substituents in diveratrole destabilize the 4,4′ methoxy groups. In contrast to this, 3,3′-dimethoxybiphenyl is stable against cleavage even to potassium in 1,2-dimethoxyethane although this system is the one among those commonly used for generation of anion radicals, which most strongly favours cleavage and replacement with hydrogen.[8] The formation of the same anion radical from both 3,3′-dimethoxybiphenyl and diveratrole as a result of the reduction with alkali metal, was established by the identical electron resonance spectra obtained in both cases.

On opening the samples to the air the deep green colour faded, and the contents was analyzed by gas-liquid chromatography and mass spectrometry. In the sample tubes where diveratrole was used as parent compound, unreacted starting material was always present, but the main reaction product was a compound with the same retention time and molecular weight as 3,3′-dimethoxybiphenyl. The amount of 3,3′-dimethoxybiphenyl formed increased with the reaction time employed (see next section). Thus, it can be concluded, that the anion radical of 3,3′-dimethoxybiphenyl is responsible for the recorded spectra in both cases.

The electron resonance spectra were recorded under different conditions in different solvents, using both sodium and potassium metal as reducing agents at three different temperatures, namely −40°, 25° and 38°C. The spectrum obtained at 25°C is shown in figure 1a, and the spectrum recorded at −40°C in figure 1b. In figure 1c the −40° spectrum is shown under conditions permitting better resolution. The main lines of the spectra may be accounted for by three sets of two protons interacting with the unpaired spin. This means that the coupling with the methoxy protons and with one pair of protons in a certain position in



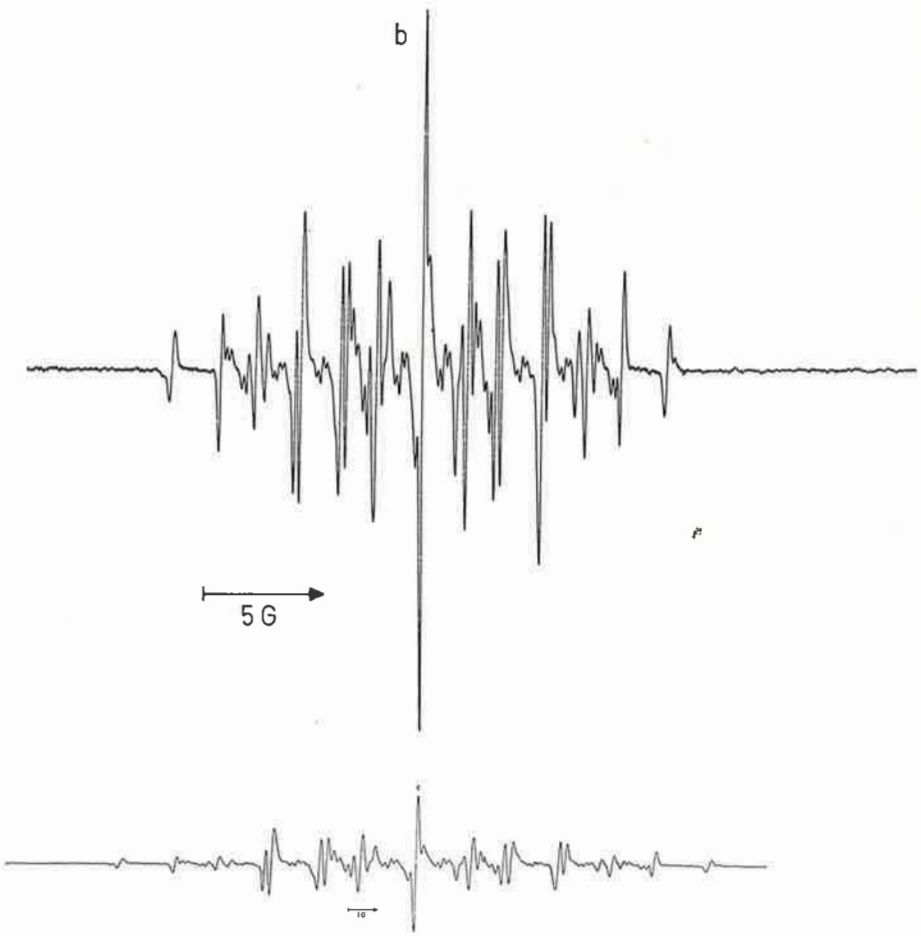


Figure 1. The electron resonance spectrum of the anion radical of 3,3′-dimethoxybiphenyl in 1,2-dimethoxyethane at different temperatures. a. 25°, b. −40° and c. the spectrum obtained at −40°C under conditions permitting better resolution.

the rings are small compared to the line width. From the observed linewidths one may conclude, that the splittings due to these protons can not exceed 0.02 G.

The triplet patterns resulting from the three sets of two protons are easily recognized, and the coupling constants at 25°C in 1,2-dimethoxyethane solution are measured to be $a_1 = 4{,}76$ G, $a_2 = 3.22$ G and $a_3 = 1.85$ G. Computer simulations

using these coupling constants, the value 0.12 G for the line widths, and a pure Lorentzian line shape function shows that this set of coupling constants very well accounts for the main features of the room temperature spectrum. The simulated spectrum is shown in figure 2a.

However, further examination of the spectra shows that different hyperfine components have different widths, and also that the intensity of the central peak consideraly exceeds the intensity predicted theoretically starting from the intensities of the other hyperfine lines. In addition, several low intensity peaks in the spectra can not be accounted for by the proposed coupling constants. These discrepancies can be understood if the effect of *cis-trans* isomerism is inwoked, or if the spectrum is considered as a superposition of the spectra of two or more chemically different species.

Cis-Trans Isomerism

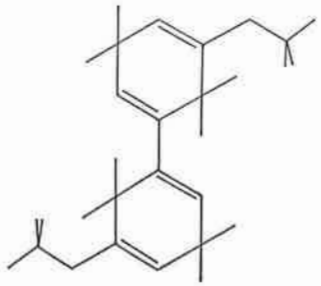
The phenomenon of *cis-trans* isomerism has been observed previously in the electron resonance spectra of numerous compounds having restricted internal rotors,[1,3,11–21] and its influence upon the observed spectra is well understood. The effect on the electron resonance spectrum can be related to the average lifetime of the contributing species, provided the molecule spends most of its time in the *cis* or in the *trans* conformation, and very little time in the transition region. If the lifetime of the isomers is larger than the reciprocal of the frequency difference between their hyperfine components, the superimposed spectra of the different species is observed. At intermediate values of the lifetime the phenomenon of linewidth alternation is observed.

The compound 3,3′-dimethoxybiphenyl has several internal degrees of freedom. The methoxy groups perform torsional oscillations around the O-C_{ar} bond, and the phenyl groups torsional oscillations around the coannular C-C bond. In principle then, the molecule can have three different *cis* and three different *trans* conformations, two of them having the methoxy groups symmetrically arranged in both phenyl groups. It is not yet conclusively established whether the methoxy groups in aromatic compounds lie in the plane of the ring or not[22], but the equilibrium mixture should nevertheless contain all these conformations, not necessarily in equal amounts.

However, before the spectrum is interpreted in terms of *cis-trans* isomerism, the possibility of the occurrence of one or several chemically different species in the reaction mixture must be critically examined. The sample medium is very reactive, and several types of reactions, such as the Birch type reduction of the radical species initially formed,[23] cleavages and rearrange-

ments,[8] ring metalation [24] and reactions between ring metalated products [7,24,25] and reactions with the solvent may occur.

A series of experiments was therefore undertaken, with reaction times ranging from 30 minutes to one week. At the end of the reaction time the electron resonance spectra of the samples were recorded, and the contents in the sample tubes analyzed by gas-liquid chromatography and mass spectrometry. In all samples the starting material 3,3′-dimethoxybiphenyl was the main component, but with a reaction time of one week 27 new compounds appeared, most of them however only in very minor amounts. With shorter reaction times most of them was not formed in detectable amounts, but two of them, hereafter named I and II, persisted also in the samples where the shortest reaction time was used. Compound I, with slightly shorter retention time than the parent compound, had the molecular weight 218 compared to 214 for the starting material. This probably corresponds to the hydrogenated product tetrahydrodimethoxybiphenyl. According to the empirical rules given by Birch and



I

Nasipuri [23] the structure I is proposed for the compound. Compound II, with slightly longer retention time than the starting material, had the molecular weight 228. This is probably a result of a reaction with the solvent. The compounds I and II were formed in approximately equal amounts, the yields ranging from 3,5 % to 26.4 % of the starting material, depending on the reaction time.

When diveratrole was used as starting material the compounds I and II could also be identified together with 3,3′-dimethoxybiphenyl and unreacted diveratrole, but also several other compounds, probably reaction products of diveratrole were present.

Because the intensities of the small peaks in the electron resonance spectra were constant, regardless of the reaction time

employed, but showed variations with the temperature, it is proposed that the spectrum is the superimposed spectra of the *cis* and *trans* conformations.

The assignment of the low intensity spectrum to the proper conformation then has to be done. It is known that small changes in molecular shape can produce large changes in the internal friction constant.[26] The shape of the *cis* conformations are such that they are likely to produce the higher value of the internal friction constant, and the *cis* conformations should therefore be energetically less favourable than the *trans* con-

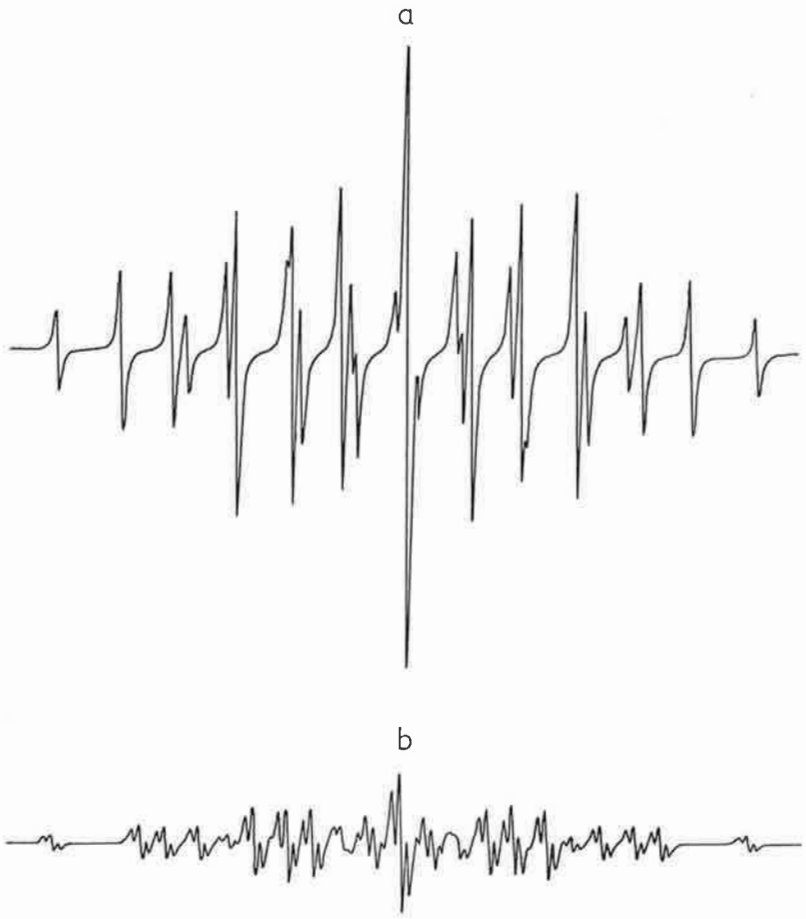


Figure 2. Simulated spectra of a. the *trans* conformation, b. the *cis* conformation. The simulation was performed using the parameters given in the text.

formations. Accordingly the low intensity spectrum is attributed to the *cis* conformations.

In the *cis* spectrum the smallest separation between the hyperfine components is 0.20 G. It is therefore assumed that a fourth set of two protons with this value of the coupling constant, unresolved in the spectrum of the *trans* conformation, interact with the unpaired spin in the *cis* conformations. Computer simulations using the coupling constants $a_1 = 4.15$ G, $a_2 = 3.20$ G, $a_3 = 2.50$ G and $a_4 = 0.20$ G give the best fit with the cis spectrum at room temperature. This simulated spectrum is shown in figure 2b. A further simulation of the two component spectrum of the *cis* and *trans* isomers result in a spectrum which well accounts for all the observed features of the recorded spectrum.

The population ratio between the two isomers was found by comparision of the intensity of the center line with its theoretically calculated intensity, starting from the intensity of the fourth of the stronger lines in the recorded spectrum. This line is a pure line from the *trans* spectrum with no intensity from the *cis* spectrum mixed in. The population ratio P_{trans}/P_{cis} thus obtained is 2.33, and for the conversion trans → cis is $\triangle G_{298} = 2.00$ kJ/mole (0.5 kcal/mole). The population ratio shows the expected temperature dependence.

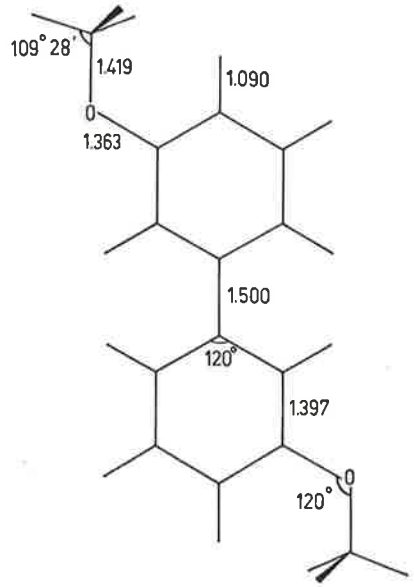


Figure 3. Bond lengths (Å) and bond angles in 3,3′-dimethoxybiphenyl. The angle between the planes of the aromatic rings was taken to be 19°.

A tentative assignment of proton hyperfine coupling constants to specific positions in the molecule was based on INDO-MO calculations of the spin densities.[27] After annihilation of the quartet state contaminant [28] the hyperfine coupling constants were obtained according to the relation:[28]

$$a_H = 711.25\ \varrho_{S_H S_H}$$

where $\varrho_{S_H S_H}$ are diagonal elements of the spin density matrix. The geometry of the molecule used in the present calculation is shown in figure 3. The results for the two symmetrical *trans* conformations are shown in figure 4. For the *cis* conformations no convergence was obtained. The results of this assignment is given in table 1. The agreement with the experimentally observed

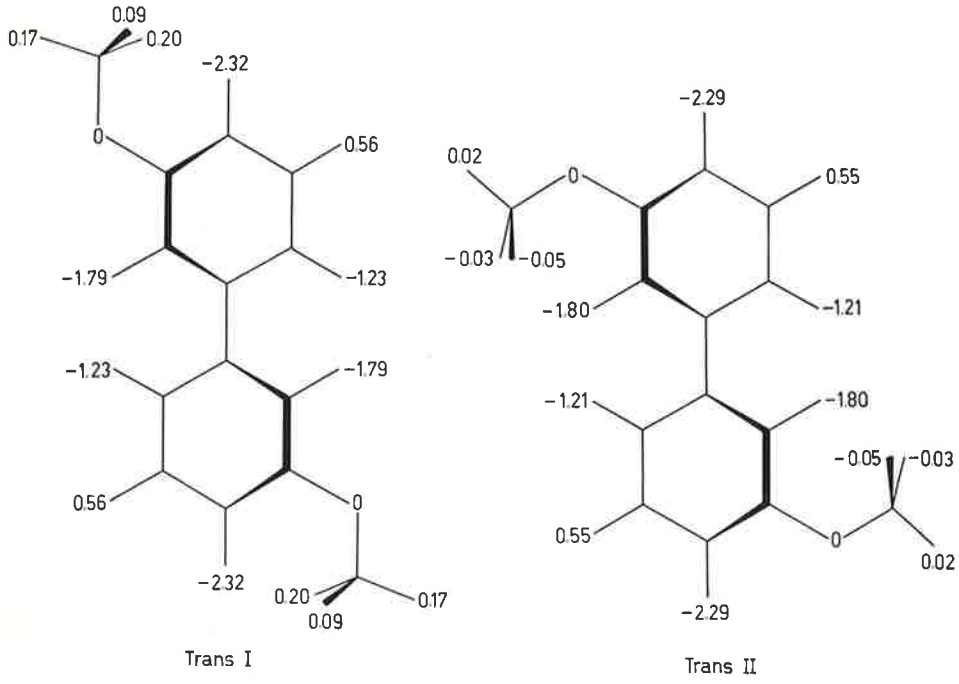


Fig. 4. The calculated proton hyperfine coupling constants in the anion radical of 3,3′-dimethoxybiphenyl. The two symmetrical trans conformations are shown. INDO with annihilation ($\langle S^2 \rangle = 0.752$).

Table 1. Comparision between the calculated (INDO MO with annihilation) and experimentally obtained proton hyperfine coupling constants. The numbering of the *trans* isomers refers to figure 4.

Position	Coupling Constants (G)			
	Calculated		Experimental	
	Trans I	Trans II	Trans	Cis
4,4′	−2,32	−2,29	4,76	4,15
2,2′	−1,79	−1.80	3.22	3.20
6,6′	−1.23	−1.21	1.85	2.50
5,5′	0.56	−0.55	Unresolved	0.20
OCH_3	0.14	0.03	»	Unresolved

coupling constants is not better than that obtained previously for other methoxy substituted aromatic compounds.[1,3,29] It is also seen that the orientation of the methoxy group has only minor influence on the calculated hyperfine coupling constants. The same fact has also been observed for anisole.[29]

In the temperature region examined in the present study, no major changes in the recorded spectra occurred, only minor changes in the coupling constants and the population ratio between the *cis* and *trans* isomers could be noted. This indicates, that the reorientation around the coannular C-C bond in the temperature interval −40° to 38°C is always slow compared to the difference in hyperfine frequency, i.e. we are observing the slow exchange region. This represents a marked difference to the cation radical of 4,4′-dimethoxybiphenyl. This cation radical has been studied in the temperature region −60° to 70°C, and the experimental results show [1,2] that in this case we are gradually coming into the region of intermediate exchange rates at temperatures below 10°C.

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References

1. Forbes, W. F. and Sullivan, P. D. *Can. J. Chem.* **46** (1968) 325.
2. Tylli, H. *Finska Kemistsamf. Medd.* **81** (1972) 24.
3. Tylli, H. *Finska Kemistsamf. Medd.* **81** (1972) 0000.
4. Seer, C. and Karl, E. *Monatsh.* **34** (1913) 631.
5. Poole, C. P., Jr. *Electron Spin Resonance*, Interscience, New York 1967.
6. Morton, A. A. and Lanpher, E. J. *J. Org. Chem.* **23** (1958) 1636.
7. Morton, A. A. and Lanpher, E. J. *J. Org. Chem.* **23** (1958) 1639.
8. Eargle, D. H., Jr. *J. Org. Chem.* **28** (1963) 1703.

9a. Nolfi, G. J., Jr. *Electron Spin Resonance Studies of Organic Free Radical Species.* Ph.D. Thesis, Massachusetts Institute of Technology 1965, p. 54–60 and references cited therein.

9b. Lehman, P. A. *Electron Spin Resonance Studies of Electronic Properties in Substituted Benzenes.* B.Sc. Thesis, Massachusetts Institute of Technology 1966.

10. Bowers, K. W. *Orbital Degeneracy in Benzene and Substituent Effects*, in Kaiser, E. T. and Kevan, L. (Ed.) *Radical Ions*, Interscience, New York 1968, p. 232–244 and references cited therein.
11. Maki, A. H. *J. Chem. Phys.* **35** (1961) 761.
12. Maki, A. H. and Geske, D. H. *J. Am. Chem. Soc.* **83** (1961) 1852.
13. Stone, E. W. and Maki, A. H. *J. Chem. Phys.* **38** (1963) 1999.
14. Bolton, J. R., Carrington, A. and Todd, P. F. *Mol. Phys.* **6** (1963) 169.
15. Forbes, F. W. and Sullivan, P. D. *Can. J. Chem.* **44** (1966) 1501.
16. Sullivan, P. D. *J. Am. Chem. Soc.* **89** (1967) 4294.
17. Barabas, A. B., Forbes, F. W. and Sullivan, P. D. *Can. J. Chem.* **45** (1967) 267.
18. Forbes, W. F. and Sullivan, P. D. *Can. J. Chem.* **46** (1968) 317.
19. Bolton, J. R. and Carrington, A. *Mol. Phys.* **5** (1962) 161.
20. Carrington, A. *Mol. Phys.* **5** (1962) 425.
21. For two useful review articles, see:
    a. Geske, D. H. *Progr. Phys. Org. Chem.* **4** (1967) 125.
    b. Hudson, A. and Luckhurst, G. R. *Chem. Rev.* **69** (1969) 191.
22. Tylli, H. *Finska Kemistsamf. Medd.* **81** (1972) 19, and references cited therein.
23. Birch, A. J. and Nasipuri, D. *Tetrahedron Letters* **6** (*1959*) 148.
24. Ehrhart, G. *Ber.* **96** (1963) 2042.
25. Ehrhart, G. *Ber.* **97** (1964) 74.
26. Vaughan, W. E., Purcell, W. P. and Smyth, C. P. *J. Am. Chem. Soc.* **83** (1961) 571.
27. Pople, J. A. and Beveridge, D. L. *Approximate Molecular Orbital Theory*, McGraw-Hill Book Company, New York 1970.
28. Beveridge, D. L. and Dobosh, P. A. *J. Chem. Phys.* **48** (1968) 5532.
29. Tylli, H. *Finska Kemistsamf. Medd.* **81** (1972) 88.

Protokoll fört vid Finska Kemistsamfundets ordinarie möte den 2. oktober 1972 kl. 19.30 på Tekniska Föreningen i Helsingfors. Ordet fördes av mag. Sjöberg och protokollet av undertecknad sekreterare. 25 medlemmar var närvarande.

§ 1. Ordförande hälsade deltagarna välkomna till höstens första ordinarie möte.

§ 2. Till protokolljusterare valdes prof. Ellfolk och FD Tötterman.

§ 3. Vid mötet den 5. april 1971, då en stadgeförändring godkändes, förbisågs valet av protokolljusterare. Mötesprotokollet från den 5.4.1971 upplästes i sin helhet och godkändes enhälligt.

§ 4. Förändringen i Finska Kemistsamfundets stadgar § 4 bör ytterligare kompletteras med meningen: Tidigare ständiga medlemmar bibehåller sina rättigheter.

§ 5. Ordförande hälsade kvällens tyska gäst dipl.ing. Preuss från Lüneburg välkomnen. Ing. Preuss talade sakkunnigt och uttömmande under rubriken: Wachse — Definition, Wachstypen, Vorkommen, Gewinnung, Raffination und Anwendung. Med anledning av föredraget yttrade sig DI v. Weissenberg, prof. Enkvist och FD Pensar.

§ 6. Kvällen avslutades med supé.

In fidem

Holger Sjöberg *Märta Lindberg*

Protokoll fört vid Finska Kemistsamfundets ordinarie möte måndagen den 6. november 1972 kl. 19.30 på Kontrollanstalten för guld och silver, Bulevarden 31, Helsingfors. Ordet fördes av FM Holger Sjöberg och protokollet av FM Märta Lindberg. Vid mötet var 51 medlemmar närvarande.

§ 1. Ordförande förklarade mötet öppnat, hälsade medlemmarna välkomna och uttryckte sin glädje över det stora deltagarantalet.

§ 2. Till protokolljusterade valdes FM Thord Bröderman och DI Kalevi Lindberg.

§ 3. Ordförande berättade i korthet om de förändringar, som planeras beträffande Acta Chemica Scandinavica. De finländska kemiska föreningarnas styrelser har godkänt ett förslag till nya stadgar för Acta Chemica Scandinavicas redaktion samt ett förslag till ändringar i stadgarna för Förlagsföreningen Acta Chemica Scandinavica. Den väsentligaste förändringen består i en uppdelning av Acta i två serier: A) Oorganisk, fysikalisk och teoretisk kemi B) Biokemi och organisk kemi. Vardera serien redigeras av en serieredaktör. För tidskriften i dess helhet utses en huvudredaktör.

På Samfundets styrelses rekommendation beslöt mötet enhälligt att överlåta äganderätten till Acta Chemica Scandinavica till takorganisationen SKKS. Ett analogt beslut har fattats på SKS' senaste möte.

§ 4. På ordförandes anmodan refererade prof. Enari planerna på fusion av de finländska kemiska tidskifterna. Planerna går ut på att sammanslå Kemian Teollisuus, Suomen Kemistilehtis A-del och FKS' Meddelandenas informationsdel. Den nya tidskriften, som går under arbetsnamnet Suomen Kemia-Finsk Kemi skall ges ut av ett förlagsaktiebolag, ägt av Kemiska Centralförbundet, SKKS, SKS, FKS och KTY. Samfundets styrelse har beslutat teckna en aktie à 500 mark i förlagsaktiebolaget, Beslutet accepterades av mötet.

Utanför fusioneringen blir Suomen Kemistilehtis B-del och FKS' Meddelandenas vetenskapliga del, som tillsammans skulle bilda en rent vetenskaplig tidning. Denna tidskrift, som skall finansieras med statsbidrag ges ut av SKKS men nyttjar förlagsaktiebolagets personal.

§ 5. Ordförande förklarade mötets officiella del avslutad och gav ordet åt kvällens föredragshållare DI Elsi Lindahl.

Fru Lindahl berättade om kontroll av guld och silver under rubriken: Probering och kontroll — nu och förr. Mötesdeltagarna visade stort intresse för ämnet, några hade t.o.m. medfört smycken av okänt guldvärde och på mindre än en minut ställde fru Lindahl »diagnos» med proberstenens hjälp. Med anledning av föredraget yttrade sig doc. Andersen, prof. Enkvist, DI v. Weissenberg, FM Holmström, prof. V. Sundman, FM Castrén, FM Ch. Waller, FM Brehmer och FM Nordström.

§ 6. Kvällen avslutades med supé på restaurang Rivoli.

In fidem

Märta Lindberg *Holger Sjöberg*

Innehåll 1972 Sisältö

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