IR SPECTRUM OF THE ZWITTERIONIC ANALOGUE
OF ZEISE'S SALT, $[H_2C\overset{(+)}{=}CHCH_2\overset{(-)}{NH_3}PtCl_3]$*

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The infrared absorption bands $(4000-200\ cm^{-1})$ of the solid title compound, precipitated in acidic media, were partially assigned. The spectrum proves that 2-propenyl-1-ammonium cation is a monodentate ligand coordinated to platinum atom through the C=C bond. Forward and back electron donation between its π-system and the central Pt atom cause a remarkable lowering in the C=C bond order. The torsional oscillation of the weakened $(C=C)_{coord}$ bond is represented by its first overtone (near $500\ cm^{-1}$) and the stretching mode of the bond by the 1349 or $1416\ cm^{-1}$ band. The "decoupled" $=CH_2$ scissoring motion is mainly characterized by a weak absorption at $1504\ cm^{-1}$. The band near $415\ cm^{-1}$ is assigned the stretching fundamental frequency of the Pt-alkene covalent bond, the three Pt—Cl bonds in $PtCl_3^{(-)}$ are characterized by their stretching fundamentals at 340, 336, and $296\ cm^{-1}$. These results were obtained with the aid of partially deuterated allylammonium ligands.

In connection with catalytic studies, we used[1] the complex *I*, $[H_2C\overset{(+)}{=}CHCH_2\overset{(-)}{NH_3}PtCl_3]$, as an efficient homogeneous catalyst for hydrosilylation of 3-amino-1-propene (further "allyl-amine"). To investigate the course of reactions between *I* and organosilicon hydrides R_3SiH, it was necessary to characterize *I* by its diagnostic IR bands. The IR spectrum of *I* was already studied by several authors[2-4], however, the published data are incomplete. Furthermore, these partial interpretations of the IR spectrum of *I* have become less reliable in the light of new facts about the structure of Zeise's salt and Zeise's dimer[5-10]. Any assignment of IR bands of *I* was made difficult by the fact that even the vibrational spectra of free allylamine were not interpreted, because the free species at room temperature is a mixture of rotamers. This complication was respected only recently[11]. The spectrum of *I* should consist of sets of the bands characteristic for $PtCl_3^{(-)}$, the $-CH_2-$ group, $-NH_3^{(+)}$, and the coordinated vinyl group. The latter represents the greatest problem for assignment.

The aim of this work was to perform a more detailed assignment of IR absorptions of *I* with the aid of partially deuterated allylamine, and to find some analytical bands of *I*.

EXPERIMENTAL

Chemicals. Commercial allylamine (Berlin-Chemie, Berlin-Adlershof, G.D.R.) was distilled before use. The IR spectra of the liquid and vapours were identical with those of the respective standards[12,13]. Allylamine hydrochloride was prepared by introducing dry HCl into a solution of allylamine in absolute ethanol. White crystals of the product were filtered off and dried under vacuum. The IR spectrum of the solid in a KCl disc agreed with the published spectrum[14], with the stipulation that two medium-to-weak bands in the latter belong to impurities (near 1 700 and 800 cm^{-1}).

Bis(allylammonium)hexachloroplatinate was prepared by mixing a solution of H_2PtCl_6 in concentrated hydrochloric acid with liquid allylamine at room temperature[15]. Yellow-orange crystals were filtered off and dried.

Complex *I*, $[H_2C{=}CHCH_2\overset{(+)}{N}H_3\overset{(-)}{P}tCl_3]$, was precipitated by the reported procedure[2] and obtained in 93% yield. Contrary to the reported results[2], *I* is slightly soluble in ethanol (96%) in which the product was dissolved (100 mg/300 ml, about 70°C). The alcohol was allowed to eva-porate slowly in air for 2 days. Light brown crystals were obtained which melted at 225—226°C with a slight darkening around 170°C (decomp. 205—220°C, ref.[2]). After the crystallization, the following absorption bands disappeared from the IR spectrum (KCl disc, in cm^{-1}): 1 969 m, 746 m, doublet 693, 702 m, 580 w, 560 w, 530 w. For $C_3H_8Cl_3NPt$ (359·5) calculated: 29·58% Cl, 3·90% N; found[16]: 29·40% Cl, 3·87% N. The N-deuterated complex, $[H_2C{=}CHCH_2\overset{(+)}{N}D_3\overset{(-)}{P}tCl_3]$, was prepared in an analogous manner[2] from allylamine and K_2PtCl_4 in an excess of DCl and D_2O in 47% yield. $H_2C{=}CHCHDNH_2$, 3-amino-1-propene-3-d_1, was obtained by reduction of acro-lein to the alcohol which in turn was transformed into the chloride and the amine: 5 g of $LiAlD_4$, suspended in 200 ml of diethyl ether, were added dropwise (30 min) to a stirred solution of 30 g of $H_2C{=}CHCHO$ in 160 ml of diethyl ether. After 1 h, 200 ml of distilled water and 160 ml of H_2SO_4 (10%) were added. The ether layer was dried by Na_2SO_4 and then the ether was distil-led off. A total of 9 g of $H_2C{=}CHCHDOH$ were obtained by rectification (b.p. 94—95°C/740 Torr; IR (liq.): a weak, broad band at 2166 cm^{-1}). To a stirred reaction mixture containing 9 g of $H_2C{=}CHCHDOH$, 13·4 g of pyridine, and 90 ml of n-heptane, a freshly distilled $SOCl_2$ (23 g) was added dropwise. After distillation and rectification, 5·4 g of $H_2C{=}CHCHDCl$ were obtained (b.p. 43·5—44°C/740 Torr, IR (liq): a weak, broad band at 2160 cm^{-1}). A total of 50 ml of liquid NH_3 and 5·4 g of the allyl chloride were placed in a pre-cooled autoclave ($-70°C$). After 4 h, the reaction temperature reached 78°C. After cooling the autoclave, the excess NH_3 was removed and the product, $H_2C{=}CHCHDNH_2$, was extracted with n-heptane (1 g; IR (solu-tion in n-heptane): a weak band at 1 841 cm^{-1} (2 × wag $={}CH_2$), a broad doublet with a shoulder at 2005 cm^{-1}, a medium band at 2 157 cm^{-1} (stretch. C—D)).

The deuterated complex, $[H_2C{=}CHCHD\overset{(+)}{N}H_3\overset{(-)}{P}tCl_3]$, was obtained by the above mentioned reaction of allylamine-d_1 with K_2PtCl_4 (42% yield). For $C_3H_7DCl_3NPt$ (360·5) calculated: 29·50% Cl, 3·88% N; found[16]: 29·25% Cl, 3·83% N.

Spectroscopic measurements. The IR absorption spectra were recorded on double-beam spectrometers Zeiss (Jena), model UR-20 and Beckman IR-7 in the 4000—200 cm^{-1} region. Solids were studied in KCl discs or Nujol suspensions (KRS-5 cell). The wavenumber scale was calibrated, the accuracy of data is ± 2 cm^{-1}, the figures are averages of 3—5 readings. The sta-tionary sample of the pressed solid *I* darkened in laser beams (Ar$^+$, He-Ne) so that no Raman spectrum was detected.

RESULTS

The IR spectrum of *I* is shown in Fig. 1 together with the wavenumbers of absorption maxima. Those which are underlined are tentatively assigned vibrational modes of zwitterion *I*, and in Table I are compared to the fundamental frequencies of solid allylamine[11], allylamine hydrochloride, and bis(allylammonium)hexachloroplatinate. The bands of the stretching vibrations of C—(H, D), N—(H, D), and Pt—Cl bonds are included in the text. A partial assignment for *I* indicates that the coordination of C=C bond to platinum atom causes remarkable changes in the electronic structure of the ligand (the C=C torsion — or twisting =CH$_2$ — is at 1005 cm^{-1} for the free species[11] and at about 500 cm^{-1} for the coordinated one). Allylammonium cation is bound to Pt atom by one covalent bond, the fundamental stretching frequency of which is near 400 cm^{-1} (*cf.*[3]).

The absorption bands of the deuterated complexes are listed below (in cm^{-1}, intensity): [H$_2$C=CHCHDNH$_3$PtCl$_3$], KCl disc and suspension in Nujol (denoted N): doublet 1581 and 1557 s, 1500 sh, doublet 1482 and 1473 s, doublet 1416 and 1401 m, 1339 m, 1309 w, ? 1284 vw, 1249 w, 1203 m, 1174 m, 1140 m, 1117 w, 1089 w, 1065 m, doublet 1033 m and 1018 w, doublet 981 w and 971 m, doublet 887 and 874 m, doublet 820 sh and 815 m, doublet 773 and 757 s, doublet 488 and 479 m, (N-doublet 491 and 482 m, N-doublet 408 sh and 401 m, N-doublet 341 and 336 s, N 295 s, N 235 br-sh.) [H$_2$C=CHCH$_2$ND$_3$PtCl$_3$], KCl disc: 1505 m, 1442 s, 1415 s, 1385 br—w, 1340 m, 1276 br—w, 1247 w, 1177 w, doublet 1152 vs and 1131 s, 1095 sh, 1023 s, 992 w, 966 m, 917 w, doublet 874 w and 859 m, 813 m, 763 s, 744 w, 713 s, doublet 485 m and 466 s, 411 s.

DISCUSSION

Let us consider first the way in which the individual bands of *I* were assigned (*cf.* Table I). Denning and Venanzi[2] quote two characteristic bands at 2060 and
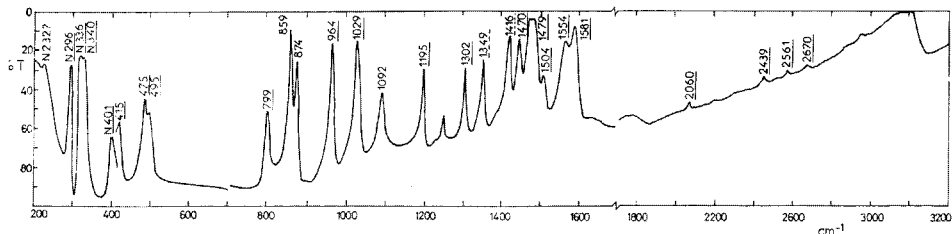

FIG. 1

IR Absorption Spectrum of Solid [H$_2$C=CHCH$_2$NH$_3$PtCl$_3$] in KCl Disc and Nujol Suspension (400—200 cm^{-1})

The wavenumbers 1246 and 1439 cm^{-1} are missing.

TABLE I

Some IR Absorption Bands of the Vinyl, Inserted $-CH_2-$, and $-NH_3^{(+)}$ Groups in Salts of Allylamine (cm^{-1})

All samples in solid state, the salts in KCl discs.

$C_3H_5NH_2{}^a$	$C_3H_5NH_3Cl$ (+) (−)	$(C_3H_5NH_3)_2$.$PtCl_6$	I^b	Assignment
1 850 w	1 900 ± ± 10 br, w	?1 880 to 1 970 br, w	?^{c}	$2 \times$ wag. $=CH_2$
1 633 m, sh	1 649 m	1 649 m	1 416 m or 1 349 m	$C=C$ stretch.
1 438 } m 1 434 }	1 433 s	1 429 s	1 504 w	$=CH_2$ sciss.
1 295 vvw	?1 325 w	1 324 br, w	?	$=CH$ i-p bend.
1 214 } vvw 1 208 }	?1 150	1 140 w, sh	?	$=CH_2$ rock.
1 005 vs	994 s	984 s	d { 495 m { 475 m	$2 \times$ $C=C$ torsion ($=CH_2$ twisting)
921 } vs 919 }	926 m	929 sh	?	$=CH_2$ wag.
643 vs	650 m } 560 w }	636 m } 554 w }	?	$=CH$ o-o-p bend.
902 } s 895 }	887 m	873 sh	?	$C-C$ stretch.
1 450 w	1 468 m	1 458 m	1 439 m	$-CH_2-$ sciss.
1 330 m	?1 375 m	1 368 w	1 302 m	$-CH_2-$ wag.
1 106 } 1 102 } vs 1 099 }	?1 110 br, m	1 094 m	1 029 s	$-CH_2-$ twist.
845 vw	870 sh	860 s	799 m	$-CH_2-$ rock.
1 060 mw	1 030 br, w	1 028 w	?	$C-N$ stretch.
1 603 } 1 599 } br, s^d 1 595 }	1 520 } s 1 510 }	1 495 sh } 1 486 s }	1 479 } s 1 470 }	NH_3^+ deg. def.
	1 603 br, s	1 582 s	1 554 } s 1 581 }	NH_3^+ sym. def.
878 } s^e 873 }	1 200 m	1 191 m	1 195 m	NH_3^+ rock.
965 } 960 } vs^f 954 } 950 }	944 s	939 s	964 m	NH_3^+ rock.
—	2 030 br, s	?1 880 to 1 970 br, w^g	2 060 w	$N-H \cdots Cl$ stretch. (+)

^{a} Frozen solid, ref.[11]; ^{b} [$H_2C=CHCH_2NH_3PtCl_3$] (+) (−); ^{c} see Discussion; ^{d} the $-NH_2$ scissoring[11]; ^{e} the $-NH_2$ wagging[11]; ^{f} the $-NH_2$ rocking[11]; ^{g} cf. $2 \times$ wag. $=CH_2$.

1 504 cm^{-1}, later[3] they added a third absorption at 417 cm^{-1} (in Nujol). The first of them was assigned the first overtone of the $=CH_2$ wagging mode, second the $(C=C)_{coord.}$ stretching, and the third one the stretching vibration of the "Pt--olefin bond".

As to the weak, broad absorption at 2 060 cm^{-1}, in this case we refuse its diagnostic value, since the band can be easily interchanged with the weak absorptions arising from the stretching vibrations of $(\overset{+}{N}-H)$ bonds associated by H-bonds. For instance, primary allylammonium salts $R\overset{(+)}{N}H_3\overset{(-)}{X}$ (X = Cl, Br, I; refs.[18-20]) and also amino acid hydrochlorides show a medium-to-strong band around 2 000 cm^{-1} followed by a group of other bands. A relevant example is allylammonium chloride (KCl disc; in cm^{-1}, intensity): 1 900 br − w, 2 030 s (half-band width 95 − 100 cm^{-1}), 2 240 br − w, 2 360 br − w, 2 406 m, 2 501 m, 2 552 m, 2 635 (maximum). Complex I exhibits the bands (in cm^{-1}): 1 980 vw, 2 060 br − w, 2 290 br − w (the others see Fig. 1). For this reason, it is then problematic to decide which band corresponds to the 2 × wag $=CH_2$ or $v(\overset{+}{N}-H)_{assoc.}$. For the N-deuterated complex, a value of 2 400 cm^{-1} represents the maximum of the broad absorption, on which smaller bands are superimposed (in cm^{-1}): 1 865 w, 1 915 w, 2 060 w, 2 140 br − w, 2 240 m, 2 275 w, 2 330 w, 2 395 s, 2 430 sh. In this case, a band of the 2 × wag $=CH_2$ can be again misinterpreted[21,22]. For similar reasons, we cannot identify with certainty the $v(C-D)$ band for the complex with the—CHD—group. It could be located in the 2 200 − 2 000 cm^{-1} region characteristic[23] for the saturated C—D bonds. The presence of deuterium atom in the zwitterion is indirectly deduced from the IR spectra of the intermediate products (*cf.* Experimental).

As to the absorption near 1 500 cm^{-1}, we do not deny its diagnostic value despite of the weak intensity. A problem is the assignment of the band. Recently, several authors[5-7] ascribed this band to two coupled vibrations involving the $=CH_2$ scissoring and the $(C=C)_{coord.}$ stretching modes. Which of the two is more responsible for this absorption, depends on the opinion of the authors[5-7]. In this case, we prefer a prevailing contribution of the $=CH_2$ scissoring mode to the 1 500 cm^{-1} band. In previous papers[8,9], we have pointed out that a strong coupling between the $=CH_2$ scissoring and the C=C stretching vibrations exists in free molecules in their electronic ground state with the planar $C=CH_2$ grouping. In complexes with the coordinated alkenes the mentioned vibrational coupling is weakened due to changes in geometry of the ligands. Owing to this fact, the two fundamental frequencies are also strongly changed, moreover, in the opposite directions. The C=C stretching fundamental (in free alkenes above 1 600 cm^{-1}) is markedly shifted down, whereas the $=CH_2$ scissoring frequency increases from about 1 400 cm^{-1} (in free species) to about 1 500 cm^{-1} after coordination (*cf.*[7]). Under these circumstances, it is necessary to find the band of $v(C=C)_{coord.}$ in the spectrum of I below 1 500 cm^{-1}. In another work[10], we have

proved that the location of this band for different alkenes is variable, depending on the number of substituents attached to the C=C bond (ethene ~1240, propene ~1360, both *cis-* and *trans-*2-butene ~1430 cm^{-1}) and on the length of the substituent (propene ~1360, 1-butene ~1375, 1-decene ~1395 cm^{-1}). Allylammonium cation represents the case of monosubstituted double bond, however, an eventual influence of the —NH$_3^{(+)}$ group should also be considered. On this basis, we assume a wider interval, 1450—1300 cm^{-1}, for possible appearance of the v(C=C)$_{coord.}$ frequency. In Fig. 1 are seen the four bands near 1450, 1420, 1350, and 1300 cm^{-1}. None of them belongs to deformation vibrations of the —NH$_3^{(+)}$ group (see Table I). The bands at 1450 and 1300 cm^{-1} are assigned by us the scissoring and wagging modes, respectively, of the inserted —CH$_2$— aliphatic group, since they are sensitive to the isotopic substitution (the case of —CHD— group). The remaining bands at 1420 and 1350 cm^{-1} do not shift and, therefore, one of them could be assigned the searched v(C=C)$_{coord.}$ frequency.

A close relation between the value of v(C=C)$_{coord.}$ and the spacing of vibrational structure of the photoelectron (PE) band from the π orbital of free alkenes was found[10]. The PE spectrum of allylamine has been already measured[24]. However, details about the corresponding vibrational strucure (of the 2nd I.P. in this case) are not mentioned[24]. An analogy with the PE spectra of allyl halides[25,26] also cannot be used, because of disagreement about the vibrational structures. Worrell[25] found the following values of v(C=C)* in cm^{-1}: ~1200 (F), ~1300 (Cl, Br) ,~1450 (I); another paper[26] quotes the separation of about 1450 cm^{-1} for allyl fluoride, the data for the other halides were not again mentioned.

Moreover, a slightly non-linear correlation between the first ionization potential of free alkenes and $\Delta v_{C=C}$ (the free minus the coordinated) was also empirically[10] found. The second ionization potential of allylamine (from π), 10·04 eV, corresponds to 1315 cm^{-1} of v(C=C)$_{coord.}$ for allylammonium ligand. However, the value of the ionization potential is influenced by an interaction between the π electrons and the lone electron pair of nitrogen atom. Therefore, we are not able to assign one of the two bands near 1420 and 1300 cm^{-1} unambiguously to v(C=C)$_{coord.}$ in the IR spectrum of *I*.

As to the third diagnostic band of *I* at 417 cm^{-1} in Nujol[3] (this work 415 cm^{-1} in KCl disc), we confirm the original assignment. It is the stretching vibration of Pt-alkene bond[3]. Let us proceed to other characteristic IR absorptions of *I*.

Firstly, it is a doublet at 475 (strong) and 495 cm^{-1} (medium) which also is not influenced by the deuterations. In accordance to the previous papers[8-10], we assign it the first overtone of the torsion (twisting) of the (C=C)$_{coord.}$ bond, the order of which is strongly reduced from 2 to about 1·5 (3 instead of 4 bonding electrons, *cf.*[27]).

Secondly, these are three strong bands around 300 cm^{-1}: 296 cm^{-1} and a doublet at 336 and 340 cm^{-1}, representing the stretching fundamentals of Pt—Cl bonds. Appearance of the three bands implies the presence of three chlorine atoms in the zwitterion, *i.e.*, it excludes the possibility that allylamine in *I* operates as bidentate ligand interacting with one Pt atom through the C=C bond and —NH$_2$ group simultaneously. It also accords with the elemental analysis of *I* proving the Cl : N ratio of 3 : 1 and not 2 : 1 (see Experimental).

From these findings for *I*, similar conclusions for other complexes can be drawn. Hartley and Wagner[28] studied the IR spectrum of the palladium analogue of *I*, $[\text{H}_2\text{C}{=}\text{CHCH}_2\overset{(+)}{\text{N}}\text{H}_3\overset{(-)}{\text{PdCl}}_3]$. The vibrational coupling of $v(\text{C}{=}\text{C})_{\text{coord}}$ and the $={=}\text{CH}_2$ scissoring modes was considered[28] and both coupled vibrations were identified by means of the 1 536 and $1 270 \text{ cm}^{-1}$ bands, respectively. A larger contribution of $v(\text{C}{=}\text{C})_{\text{coord}}$ to the $1 536 \text{ cm}^{-1}$ absorption and of the $={=}\text{CH}_2$ scissoring to the 1 270 cm^{-1} was prefered[28] ("case *a*"), the reverse assignment was refused ("case *b*"). On discussing a relation between the $={=}\text{CH}_2$ scissoring frequency in the free and coordinated allylamines, the authors[28] have chosen an absorption at $1 295 \text{ cm}^{-1}$ as a counterpart of the $1 270 \text{ cm}^{-1}$ band in the complex. Unfortunately, this fundamental frequency of the free allylamine belongs to another vibration, the in-plane deformation of the lone $={=}\text{C}{-}\text{H}$ bond $(v_{13}$, vinyl hydrogen i/p def. in ref.[11], *cf.* Table I), whereas the $={=}\text{CH}_2$ scissoring mode of the free molecule absorbs near $1 430 \text{ cm}^{-1}$ $(v_{11}$ in ref.[11], *cf.* Table I), in accordance with all known vinyl and allyl derivatives (*e.g.*[29]).

a   *b*
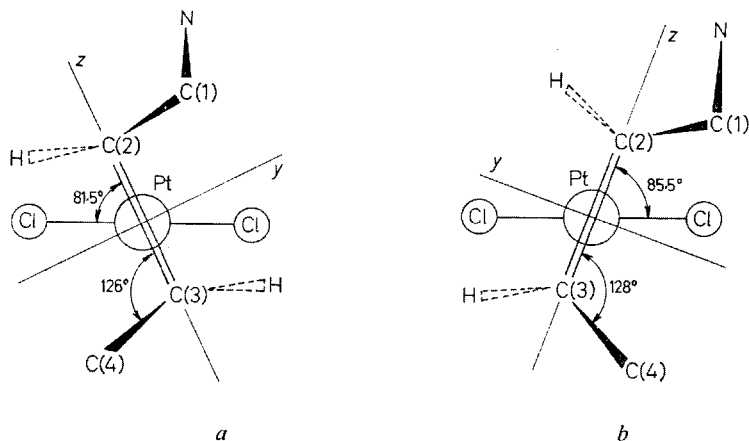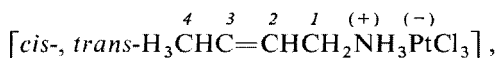
FIG. 2

Projections along the Pt-Alkenyl Bond of 2-Butenylammonium Complexes with the Configurations *a*) *trans* [C(2)R : C(3)R] and *b*) *cis* [C(2)R : C(3)S]

   The dashed lines express the uncertainty in locations of hydrogen atoms.

So, in our opinion the "case b" seems to be more probable, *i.e.*, $v(C{=}C)_{coord.}$ should be located at wavenumbers lower than $1\,500\ cm^{-1}$ but higher than about $1\,270\ cm^{-1}$.

Although I was not studied crystallographically, the configuration of the skeleton of allylammonium cation can be deduced from the X-ray diffraction data for homologous internal salts[30]

$$\overset{4}{}\ \overset{3}{}\ \overset{2}{}\ \overset{1}{}\ \overset{(+)}{}\ \overset{(-)}{}$$
$$\left[cis\text{-},\ trans\text{-}H_3CHC{=}CHCH_2NH_3PtCl_3\right],$$

the structures of which are shown in Fig. 2. It is seen that the distorsions of the ligands inhibit the replacement of the adjacent chlorine atom by nitrogen atom of the $-NH_2$ group of allylamine. Although not bonded in a PtC_2 ring[30], the atoms C (2), Pt, and C (3) form a scalene triangle, the plane of which is not perpendicular to the $PtCl_3$ plane, the same holds for the $C(2)C(3)$ bond. From this point of view, it is understandable why the lengths of three Pt—Cl bonds are not equivalent. Such an irregular configuration of all the nuclei in these complexes indirectly explains the enlarged number of bands in the spectrum of our monodeuterated zwitterion with the
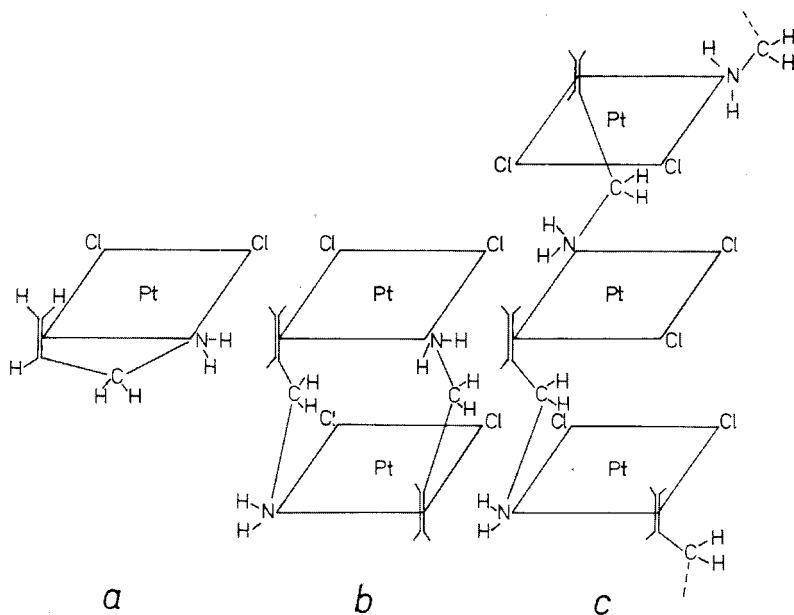

FIG. 3

Complexes of Allylamine with Pt(II) in *a*) Acidic and *b*, *c*) Neutral Media

   a) Bidentate ligand in monomer, *b*) bidentate ligand in dimer, *c*) bidentate ligand in oligomer

—CHD— group. The C—D bond is oriented in two ways with respect to the $PtCl_3$ plane, *i.e.*, the influence of the surrounding on it is dissimilar. For this reason, we were not able to interpret the spectrum completely. Clearly, the zwitterions containing —CD_2— and/or =CD_2 groups, are needed for further studies[17].

It should be pointed out that the facts discussed here are related to the complex *I* which is formed in acidic media, in this case in aqueous HCl. If precipitation of a complex consisting of allylamine and platinum(II) is carried out by neutralization of the solution by an alkali hydroxide, the coordination of —NH_2 group of allylamine to Pt atom is possible, however, in intermolecular (Fig. 3*b*, *c*), not intramolecular way. Kotov and Essen[4] proposed the only possiblity, formation of the dimer (Fig. 3*b*); however, an oligomerization seems to us equally probable (Fig. 3*c*). The evidence published thus far[4], *i.e.*, the elemental analysis, cannot solve this ambiguity.

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Note added in proof : The vibrational spectra of 5 deuterated allylamines[17] were interpreted[31].

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