Infrared Spectra of Succinimide and Maleimide in the Crystalline State

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Although the infrared spectra of succinimide[1-4] and maleimide[5,6] have been discussed in many papers, no investigation concerning the spectral change on deuteration has been made for these compounds. In a series of studies on the infrared spectra of acyclic imides[7-10], we have given assignments to the characteristic frequencies of the trans-trans -CONHCO- group. In contrast with the case of acyclic imides, the -CONHCO- group in cyclic imides is expected to be nearly in the cis-cis configuration. Under these circumstances, the present paper deals with succinimide and maleimide with a view of discussing the characteristic frequencies of the cis-cis -CONH·CO- group. The infrared spectra of both the undeuterated and the *N*-deuterated species of these compounds in the crystalline state have been measured in the region 4000 to 400 cm^{-1}, and several bands have been found to result from the vibrations of the -CONHCO- group. The spectral change on the addition of potassium iodide has also been examined for succinimide and succinimide-*d*.

Results and Discussion

Molecular Structure and Infrared Spectra.— By X-ray diffraction study, the molecular structure of succinimide in the crystalline state has been established to be planar except for the positions of the hydrogen atoms[11]. Furthermore, the sum of the three interbond angles around the nitrogen atom has been calculated to be 358°. Thus, in the following discussions, the cis-cis configuration of the -CONHCO- group will be adopted as a good approximation for succinimide. Although the molecular structure of maleimide has not been determined crystallographically, it may be reasonable to assume that the -CONHCO- group of this molecule in the crystalline state is also nearly in the cis-cis configuration.

Thus, the most symmetric arrangement of atoms in the molecule affords the point group C_{2v}, although the actual molecules may be of a somewhat lower symmetry. In fact, according to Mason[11] and Mirone[5], the crystals of succinimide and of maleimide consist of cyclic dimers, each of which involves two equivalent hydrogen bonds, so a molecule contains botb

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[2] H. Lenormant, *Bull. soc. chim. France*, (5) **15**, 33 (1948).

[3] J. Chouteau, ibid., (5) **20**, 1148 (1953).

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[7] T. Uno and K. Machida, This Bulletin, **34**, 545 (1961).

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[9] T. Uno and K. Machida, ibid., **34**, 821 (1961).

[10] T. Uno, K. Machida and I. Hamanaka, ibid., **34**, 1448 (1961).

[11] R. Mason, *Acta Cryst.*, **9**, 405 (1956).

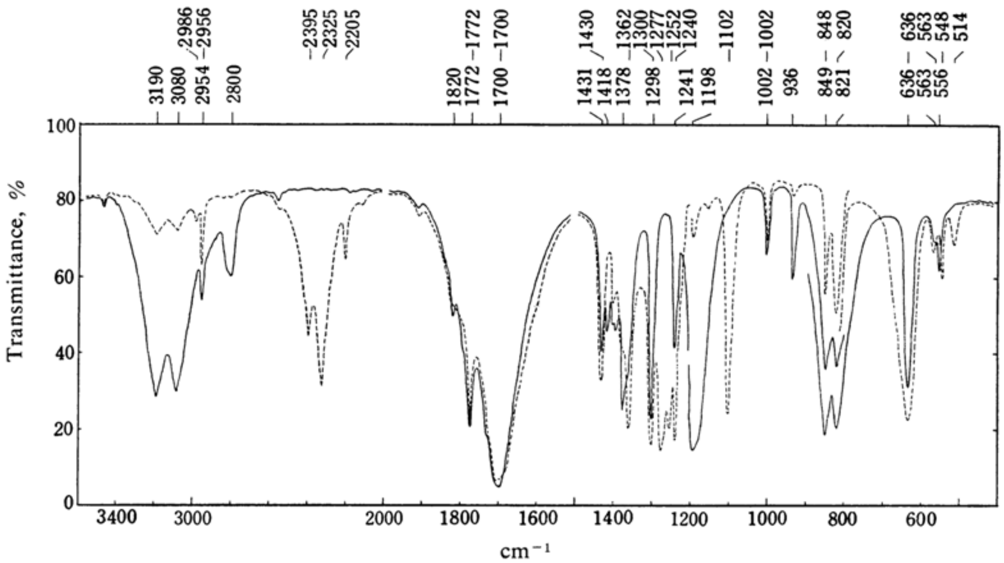

Fig. 1.* Infrared spectra of succinimide (solid line) and succinimide-*d* (broken line).

* ($3600\sim2000\ \mathrm{cm}^{-1}$ and $1500\sim1200\ \mathrm{cm}^{-1}$) hexachlorobutadiene pastes; ($2000\sim1500\ \mathrm{cm}^{-1}$ and $1200\sim400\ \mathrm{cm}^{-1}$) Nujol pastes.

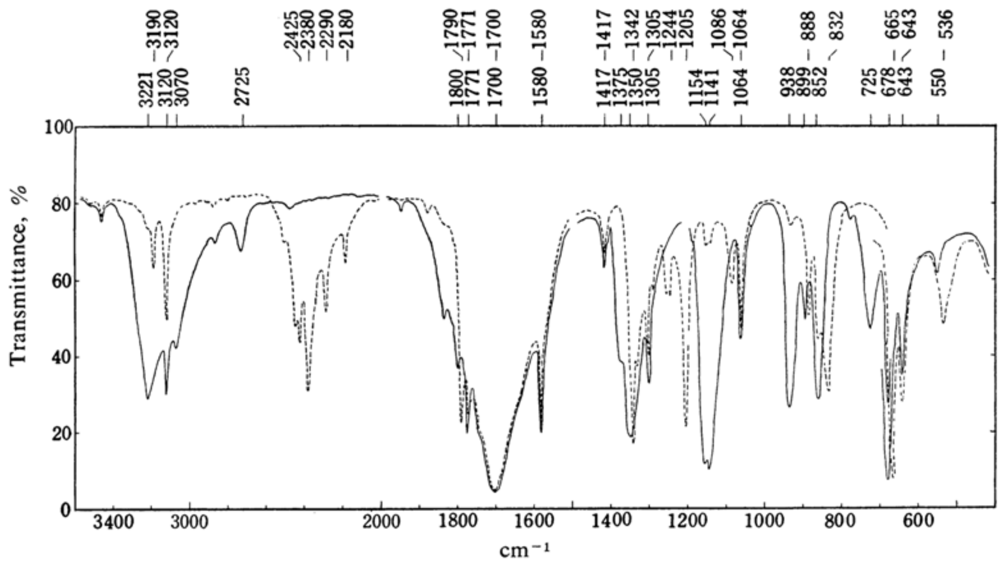

Fig. 2.* Infrared spectra of maleimide (solid line) and maleimide-*d* (broken line).

* ($3600\sim2000\ \mathrm{cm}^{-1}$ and $1500\sim1200\ \mathrm{cm}^{-1}$) hexachlorobutadiene pastes; ($2000\sim1500\ \mathrm{cm}^{-1}$ and $1200\sim400\ \mathrm{cm}^{-1}$) Nujol pastes.

bonded and free C=O bonds. This means both that the two C=O bonds in the –CONHCO– group are not equivalent to each other in the crystalline state and that the molecular symmetry of these compounds deviates appreciably from C_{2v}. This deviation from C_{2v} may or may not affect the feature of an absorption band according to the nature of vibration related to the band under consideration. The infrared spectra of succinimide and succinimide-*d* are shown in Fig. 1, while those of maleimide and maleimide-*d* are shown in Fig. 2. Table I shows the characteristic frequencies of succinimide and succinimide-*d*, together with their assignments. Similarly, Table II is concerned with maleimide and maleimide-*d*. The non-characteristic frequencies of these compounds are listed in Tables IV and V. In the region below $1100\ \mathrm{cm}^{-1}$, the infrared spectrum of maleimide in the crystalline state measured in

TABLE I. THE CHARACTERISTIC FREQUENCIES OF SUCCINIMIDE AND SUCCINIMIDE-*d* (in cm^{-1})*1

Undeut. compd.	*N*-Deut. compd.	Assignments*2	Symmetry*3 species
3190 s, br.*4		ν_{N-H}	A′
3080 s, br.		1700+1418	
2800 w		1418+1378	
	2395 w	1277+1102	
	2325 s	ν_{N-D}	
	2205 w	1102×2	
1772 m	1772 m	$\nu_{C=O}$ (Imide I) free	
1700 vs, br.	1700 vs, br.	bonded	
1418 m		$\delta_{N-H}+\nu_{C-N-C}$ (antisym)	
1378 s		ν_{C-N-C} (sym)	
	1362 s	ν_{C-N-C} (sym)	
	1277 s	ν_{C-N-C} (antisym)	
1198 s		$\delta_{N-H}+\nu_{C-N-C}$ (antisym)	
	1102 s	δ_{N-D}+skeletal	
936 m		Skeletal*5	
	820 m	δ_{N-D}+skeletal	
636 m	636 m, br.	$\delta_{C=O}$ (Imide IV)	
849 s		π_{N-H} (Imide V)	A″
	(636)*6	π_{N-D} (Imide V′)	
556 w		$\pi_{C=O}$ (Imide VI)	
	548 w	$\pi_{C=O}$ (Imide VI′)	

TABLE II. THE CHARACTERISTIC FREQUENCIES OF MALEIMIDE AND MALEIMIDE-*d* (in cm^{-1})*1

Undeut. compd.	*N*-Deut. compd.	Assignments*2	Symmetry*3 species
3221 s, br.*4		ν_{N-H}	A′
3070 sh		1700+1375	
2725 w		1375+1350	
	2425 w	1342+1086 or 1205×2	
	2380 s	ν_{N-D}	
	2290 w	1205+1086	
	2180 w	1086×2	
1771 m	1771 m	$\nu_{C=O}$ (Imide I) free	
1700 vs, br.	1700 vs, br.	bonded	
1375 sh		$\delta_{N-H}+\nu_{C-N-C}$ (antisym)	
1350 s, br.		ν_{C-N-C} (sym)	
	1342 s	ν_{C-N-C} (sym)	
	1205 s	ν_{C-N-C} (antisym)	
1154 1141 s, db.		$\delta_{N-H}+\nu_{C-N-C}$ (antisym)	
	1086 w	δ_{N-D}+skeletal	
938 s		Skeletal*5	
	832 s	δ_{N-D}+skeletal	
643 m	643 m	$\delta_{C=O}$ (Imide IV)	
725 m, br.		π_{N-H} (Imide V)	A″
	536 w, br.	π_{N-D} (Imide V′)	
550 w		$\pi_{C=O}$ (Imide VI)	

*1 The combination and the overtone frequencies above 2000 cm^{-1} are also listed here.

*2 Greek letters denote the following vibrational modes: ν, stretching; δ, in-plane deformation; π, out-of-plane deformation.

*3 The symmetry species are based on the point group C_s obtained by eliminating the C_2 axis from C_{2v}.

*4 Abbreviations indicating the absorption intensities and the band shapes are as follows: vs, very strong; s, strong; m, medium; w, weak; sh, shoulder; br., broad; db., doublet.

*5 This frequency is listed here so that the behavior on deuteration may be indicated explicitly.

*6 Parentheses show the overlapped frequencies.

the present study is somewhat different from that reported by Chiorboli and Mirone[5]. Thus, their interpretation of the absorption bands below 1100 cm^{-1} is not consistent with the results discussed below.

The In-plane Characteristic Frequencies Below 2000 cm^{-1}. — For both succinimide[1,2] and maleimide[5], the strongest band near 1700 cm^{-1} and the medium band near 1770 cm^{-1} have been assigned to the bonded and the free C=O stretching vibration respectively. Although there remains a question regarding how far the vibrational coupling contributes to the separation (ca. 70 cm^{-1}) between the two C=O stretching frequencies, no attempt will be made to discuss this problem further. The following discussion will be focussed on the absorption frequencies sensitive to the azimutal angles around the C–N bonds of the –CONHCO– group. In this respect, it is interesting to compare the infrared spectra of these cyclic imides with those of the acyclic imides reported in preceding papers[7,8]. A similarity in the characteristic bands between the trans-trans –CONHCO– and the trans –CONH– group has already been pointed out[8]. Thus, the spectral changes on deuteration for the cis-cis –CONHCO– group may be expected to be analogous with those for the cis –CONH– group. The normal coordinate analysis for the cis *N*-methylacetamide[12] has shown that the coupling between the N–H in-plane deformation and the C–N stretching vibration is less significant for the cis –CONH– group than for the trans –CONH– group. This difference in the coupling results in the characteristic frequencies of the cis –CONH– group being markedly different from those of the trans –CONH– group. The most important difference is that the former does not show the amide II band which has been well established for the latter in the region near 1550 cm^{-1}. That a similar situation holds also for the –CONHCO– group is confirmed by the fact that neither succinimide nor maleimide shows any band in the region 1600 to 1450 cm^{-1}, whereas the trans-trans and the trans-cis –CONHCO– group show the strong imide II band which disappears on deuteration[7,8]. Instead, succinimide and maleimide show a band in the region 1380 to 1340 cm^{-1} which undergoes a low-frequency shift by 8~16 cm^{-1} on deuteration. A similar spectral change on deuteration has been reported for monosubstituted amides containing the cis –CONH– group[12]. As a consequence of these spectral features, the cis-cis –CONHCO– group is expected to show two fundamentals due to the C–N–C stretching vibrations in the region 1500 to 1000 cm^{-1}. Since it is quite probable that a marked coupling occurs among the stretching vibrations of adjacent bonds with similar bond orders, one of these vibrations may be more symmetric or less antisymmetric in nature than the other vibration. Analogy with the cis –CONH– group suggests that the band in the region 1380 to 1340 cm^{-1} observed for both the undeuterated and the *N*-deuterated species of succinimide and maleimide results essentially from the symmetric C–N–C stretching vibration. On the other hand, the antisymmetric C–N–C stretching vibration of the undeuterated species seems to be involved in an appreciable coupling with the N–H in-plane deformation vibration. This is suggested from the fact that two absorptions, which disappear on deuteration, are observed in the region 1450 to 1100 cm^{-1} for both of these cyclic imides. With respect to succinimide, these are the medium but well-resolved band at 1418 cm^{-1} and the strong and broad band near 1200 cm^{-1}. The high- and low-frequency counterparts of maleimide are observed at 1375 cm^{-1} as a shoulder of the broad symmetric C–N–C stretching band and near 1150 cm^{-1} as a strong doublet (1154 and 1141 cm^{-1}), respectively. These bands are considered to result from coupling between the N–H in-plane deformation and the antisymmetric C–N–C stretching vibration. Although the high-frequency bands are not so strong, the assignment to an in-plane vibration within the –CONHCO– group is confirmed, as will be discussed later, by an examination of the combination tone near the N–H stretching frequency. It is expected that the *N*-deuterated species will show a band contributed mainly from the antisymmetric C–N–C stretching vibration which is involved in the coupling for the undeuterated species. The bands at 1277 and 1205 cm^{-1} observed for succinimide-*d* and maleimide-*d* respectively are assigned to this vibration, so the product rule holds well. The other bands appearing on deuteration in this region may be considered not to be fundamentals.

The N–D in-plane deformation vibration has been reported to couple with other vibrations for the cis –CONH– group[12]. This situation seems to hold also for cyclic imides. The strong band at 1102 cm^{-1} for succinimide-*d* and the weak band at 1086 cm^{-1} for maleimide-*d* are undoubtedly contributed from the N–D in-plane deformation vibration. These frequencies, however, in comparison with the N–D in-plane deformation (imide III′) frequencies of the trans-trans –CONDCO– group (ca. 960 cm^{-1}), seem to be so high that they can only with difficulty be assigned to the pure N–D

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[12] T. Miyazawa, *J. Mol. Spectroscopy*, **4**, 155 (1960).

in-plane deformation vibration. Thus, an appreciable coupling with other vibrations is expected to be the case with these frequencies. This is confirmed by the fact that the band near 936 cm^{-1} (medium for succinimide and strong for maleimide) of the undeuterated species disappears on deuteration. The alternative frequencies which satisfy the product rule can be found at 820 and 832 cm^{-1} for succinimide-*d* and maleimide-*d* respectively. These bands may also be contributed from the N–D in-plane deformation vibration. The marked difference in intensity in the corresponding bands between succinimide-*d* and maleimide-*d* may be due to the structural difference in the hydrocarbon residues of these two compounds.

The band near 640 cm^{-1} observed for both succinimide and maleimide remains unchanged on deuteration. Comparison with the other related compounds leads to the assigment of this band to the C=O in-plane deformation (the imide IV) vibration. Thus, nearly complete assignments have been made for the characteristic in-plane vibrations of succinimide, maleimide and their *N*-deuterated compounds. It is seen that succinimide and maleimide show quite analogous frequencies as far as the characteristic vibrations are concerned.

The Out-of-plane Characteristic Frequencies.—The broad bands at 849 and 725 bm^{-1}, observed respectively for succinimide and maleimide, disappear on deuteration, suggesting that they are due mainly to the N–H out-of-plane deformation (imide V) vibration. As alternative N–D out-of-plane deformation (imide V′) frequencies, the band at 636 cm^{-1} of succinimide-*d* and the band at 536 cm^{-1} of maleimide-*d* are quite probable. The sharp medium band at 556 cm^{-1} observed for succinimide shifts to 548 cm^{-1} on deuteration. These bands may be due to the C=O out-of-plane deformation (imide VI and VI′) vibration. The corresponding band for maleimide may be that observed at 550 cm^{-1}. However, this band's shift on deuteration is not obvious owing to the overlapping of the broad imide V′ band at 536 cm^{-1}. The marked differences in the imide V and V′ frequencies between succinimide and maleimide suggest that there is an appreciable difference in the strength of the hydrogen bond between the crystals of these compounds. This is consistent with the information from the N–H and N–D stretching frequencies, which will be discussed later. It is to be noticed here that similar differences in the N–H and N–D out-of-plane deformation frequencies have been pointed out also for monosubstituted amides[13] and acyclic imides[10].

The Product Rule.—The theoretical product rule ratios for species A′ and A″ are calculated from the principal moments of inertia, which, in turn, may be calculated for succinimide from Mason's data on the molecular structure[11]. On the other hand, the theoretical ratio for the frequencies above 2000 cm^{-1} may be given by $(\mu_N+\mu_H)^{1/2}/(\mu_N+\mu_D)^{1/2}$, since in this case only the N–H and the N–D stretching frequencies are involved. This ratio, together with the above-obtained ratio for all A′ frequencies, leads to the evaluation of the theoretical ratio for the A′ frequencies of succinimide below 2000 cm^{-1}. Table III shows these three theoretical ratios, together with the experimental ratios obtained from the frequencies which undergo an appreciable shift on deuteration. The theoretical ratios for maleimide are expected to be not far from the corresponding ratios for succinimide. It is seen that the observed ratios agree well with the theoretical ones. This result gives support to the assignment of the characteristic frequencies proposed in the present paper.

TABLE III. THE PRODUCT RULE RATIOS
(Undeut. compd./*N*-Deut. compd.)

	Succinimide		Maleimide	
	Theor.	Obs.*	Theor.	Obs.*
A′ (ν_{N-H}/ν_{N-D})	1.37	1.38	1.37	1.35
A′ (<2000 cm^{-1})	1.44	1.40	—	1.38
A″	1.38	1.35	—	1.38

* The frequencies used are as follows: succinimide (A′) 3190, 1418, 1378, 1198, 936, (A″) 849, 556; succinimide-*d* (A′) 2325, 1362, 1277, 1102, 820, (A″) 636, 548; maleimide (A′) 3221, 1375, 1350, 1148 (the mean value of 1154 and 1141), 938, 899, (A″) 725, 678; maleimide-*d* (A′) 2380, 1342, 1205, 1086, 888, 832, (A″) 665, 536.

The N–H and N–D Stretching Frequencies.—As seen from Figs. 1 and 2, the N–H and the N–D stretching bands show quite different features from each other for these two cyclic imides. Similar phenomena have been observed for several acyclic imides and interpreted in terms of the Fermi resonance[9]. As shown in Tables I and II, the features of the N–H and the N–D stretching bands of cyclic imides in the present study can also be well elucidated from analogous points of view. The broad doublet (3190 and 3080 cm^{-1}) of succinimide is considered to result from the Fermi resonance between the the N–H stretching vibration and the combination tone of the lower characteristic fundamentals (1700 and 1418 cm^{-1}). The sum of these fundamental frequencies

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[13] T. Miyazawa, T. Shimanouchi and S. Mizushima. *J. Chem. Phys.*, **24**, 408 (1956).

(3118 cm^{-1}) exceeds the frequency of the lower peak (3080 cm^{-1}) by about 40 cm^{-1}, suggesting that the resonance is considerable. This is consistent with the fact that the peaks at 3190 and 3080 cm^{-1} show almost the same intensity. The difference between the unperturbed and the perturbed frequency is expected to be the same for the N–H stretching frequency as for the combination frequency involved in the Fermi resonance[14]. Thus, the unperturbed N–H stretching frequency of succinimide may be estimated to be near 3150 cm^{-1}, that is, 40 cm^{-1} lower than the frequency of the higher peak (3190 cm^{-1}). Nakamoto et al. have pointed out the linear dependence of the N–H stretching frequencies on the N···O distances in the N–H···O hydrogen bond[15]. In this connection, the present result is nearly consistent with the crystallographic data, according to which the N···O distance is 2.85 Å for succinimide[11].

On the other hand, the weakness of the band at 3070 cm^{-1} of maleimide, as well as the small difference between the observed (3070 cm^{-1}) and the combined ($1700+1375=3075$ cm^{-1}) frequencies, indicates that the Fermi resonance is less significant for this molecule. This means that the unperturbed N–H stretching frequency of maleimide is not far from the observed frequency (3221 cm^{-1}). From these results it is seen that there is an appreciable difference (ca. 70 cm^{-1}) in the unperturbed N–H stretching frequencies between succinimide and maleimide. Thus, it may be suggested that the hydrogen bond involved in succinimide is stronger than that involved in maleimide. This results is consistent with the information from the spectral differences between these compounds in the potassium bromide region.

The weak peaks associated with the N–D stretching band can be interpreted as overtone or combination bands related to the characteristic frequencies in the region 1400 to 1000 cm^{-1}. This means that these characteristic frequencies of the –CONHCO– group are distinguished from the non-characteristic frequencies not only by their sensitivity to deuteration but also by their tendency to give rise to overtone or combination bands.

Frequencies Due to the Hydrocarbon Residues. —It is observed that the ring strain raises the C–H streching frequencies of these compounds by 60 to 100 cm^{-1} from the ordinary positions for the structures –CH$_2$– and =CH–[16]. However, the effect of the ring strain on the C–H deformation frequencies of these compounds is not so obvious as that on the stretching frequencies. If the symmetry C_{2v} is assumed, succinimide is expected to show six C–H deformation frequencies, since in this case the A_2 CH$_2$ twisting and the A_2 CH$_2$ rocking vibration are infrared inactive. Furthermore, the B_1 CH$_2$ bending and the A_1 CH$_2$ wagging frequencies may not be observed owing to the small transition moments for the corresponding vibrations. In fact, succinimide shows four bands, at 1431, 1298, 1241 and 1002 cm^{-1}, which remain unshifted on deuteration. These bands are assigned to the C–H deformation vibrations, as shown in Table IV. Thus, in contrast with the case of the vibrations related to the –CONHCO– group, the deviation from C_{2v} seems to be negligible with respect to the C–H deformation vibrations. Similarly, the three C–H deformation frequencies of maleimide may be assigned as shown in Table V. It should be noticed here that the C–H out-of-plane deformation frequency undergoes an appreciable shift on deuteration. The bands left unassigned so far in the region 1000 to 800 cm^{-1} may be due to the in-plane ring skeletal vibrations, to which the contribution of the stretching of the C–C bonds is significant.

TABLE IV. THE NON-CHARACTERISTIC FREQUENCIES OF SUCCINIMIDE AND SUCCINIMIDE-*d* (in cm^{-1})

Undeut. compd.	*N*-Deut. compd.	Assignments
	2986 w	ν_{CH_2}
2954 w	2956 w	
1431 m	1430 m	CH$_2$ bend.
1298 s	1300 s	CH$_2$ wag.
1241 m	1240 m	CH$_2$ twist.
1002 m	1002 m	CH$_2$ rock.
	1252 sh	?
(849)	848 m	Skeletal
821 s	(820)	

TABLE V. THE NON-CHARACTERISTIC FREQUENCIES OF MALEIMIDE AND MALEIMIDE-*d* (in cm^{-1})

Undeut. compd.	*N*-Deut. compd.	Assignments
	3190 w	ν_{C-H}
3120 s	3120 s	
1580 m	1580 m	$\nu_{C=C}$
1417 w	1417 w	δ_{C-H}
1305 m	1305 m	δ_{C-H}
	1255 1244 m, db.	?
1064 m	1064 m	Skeletal
899 m	888 m	
852 s	852 sh	
678 s	665 s	π_{C-H}

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[14] T. Miyazawa, *J. Mol. Spectroscopy*, **4**, 168 (1960).

[15] K. Nakamoto. M. Margoshes and R. E. Rundle, *J. Am. Chem. Soc.*, **77**, 6480 (1955).

[16] L. J. Bellamy, "The Infra-red Spectra of Complex Molecules", John Wiley & Sons, Inc., New York (1958), pp. 15, 34.

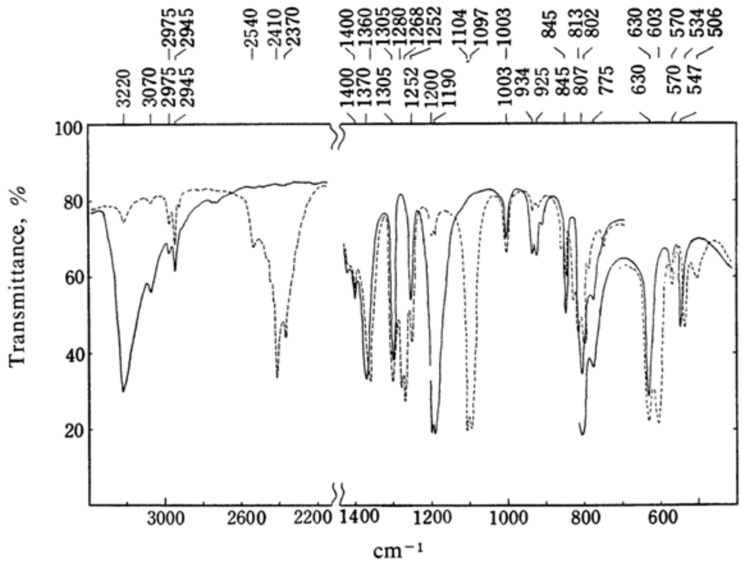

Fig. 3. Infrared spectra of succinimide (solid line) and succinimide-*d* (broken line) mixed with potassium iodide.

Interaction with Potassium Iodide.—Baker has reported that succinimide undergoes a marked spectral change on the change of matrix in the disk technique from potassium bromide to potassium iodide⁴⁾. The anomalous infrared spectrum of succinimide measured with potassium iodide disks has been given for the region 1400 to 750 cm^{-1}. We have obtained an anomalous spectrum identical with that shown in Baker's paper by using a sample obtained by removing water from an aqueous solution of succinimide and potassium iodide. This procedure can be used to obtain the anomalous spectrum of the *N*-deuterated compounds corresponding to that of the undeuterated species. These anomalous spectra are shown in Fig. 3. From this spectral change due to potassium iodide, the several interesting results following concerning the characteristic frequencies have been obtained:

(1) The high-frequency shift of the N–H (N–D) stretching band and the low-frequency shift of the imide V (V′) band are observed on the spectral change due to potassium iodide. This suggests that the N–H hydrogen atom is involved in the interaction with the iodide anion, as in the case of diacetamide¹⁰⁾. (2) The weak overtone or combination bands near the N–D stretching band also undergo a frequency shift according to the change in the lower characteristic fundamentals. (3) The overlapping of the imide IV and V′ bands at 634 cm^{-1} of the *N*-deuterated species is removed by the low-frequency shift of the latter band. Thus, it is explicitly observed that the former band remains unshifted on deuteration. (4) The band near 830 cm^{-1} observed for the *N*-deuterated species splits according to the splitting of the band at 936 cm^{-1} of the undeuterated species. This may be taken as indicating that the band at 936 cm^{-1} of the undeuterated species actually shifts to 820 cm^{-1} on deuteration. This frequency shift is not obvious in the ordinary spectra owing to the presence of the band at 820 cm^{-1} for both the undeuterated and the *N*-deuterated species. (5) The band at 1198 cm^{-1} of the undeuterated species and the band at 1102 cm^{-1} of the *N*-deuterated species split in a similar way, suggesting that these bands are contributed from vibrations of a similar nature. These results are consistent with the interpretation of the absorption bands given in the present paper, thus giving supports to the assignments made in Tables I, II, IV and V.

Experimental

Materials and Measurement.—G. P. grade succinimide was recrystallized from ethylmethylketone, m. p. 125°C. Maleimide was prepared from dimethyl maleate through maleindiamide, according to Rinkes' method¹⁷⁾, and purified by sublimation under reduced pressure, m. p. 93°C. No spectral change was observed for either of these compounds on crystallization from water. Thus, succinimide-*d* and maleimide-*d* were prepared by the usual exchange reaction with deuterium oxide. A Koken DS-301 infrared spectrophotometer equipped with rock salt or potassium bromide prisms was used. The measurements were carried out on pastes in

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17) I. J. Rinkes, *Rec. trav. chim.*, **46**, 272 (1927); **48**, 961 (1929).

Nujol or hexachlorobutadiene under the same conditions as those reported previously[7].

Interaction with Potassium Iodide.—A small amount of an aqueous solution of succinimide and potassium iodide was evaporated to dryness under reduced pressure (ca. 5 mmHg) at 80～90°C. The samples thus obtained were measured immediately as pastes. The anomalous spectrum for the *N*-deuterated species was obtained by the same procedure, using deuterium oxide instead of water.

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