

## ON PHTHALIDES AND INDANDIONES. XLI.\*

INFRARED SPECTRA AND SUBSTITUENT EFFECTS  
IN 3-ARYLMETHYLENEPHTHALIMIDINES  
AND 3-ARYLMETHYLENETHIOPHTHALIDES

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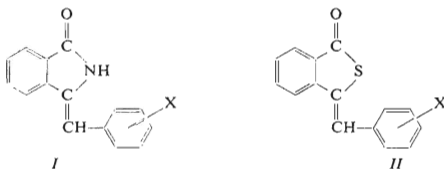
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The infrared spectra of 22 substituted derivatives of *trans*-3-phenylmethylenephthalimidine and *trans*-3-phenylmethylenethiophthalide were studied in the region of C=O, C=C, and N—H stretching vibrations. In both series of compounds linear correlations were observed between the C=O stretching frequencies and the  $\sigma$ -constants of substituents. The results were compared with those published in preceding papers. On the basis of these comparisons the influence of sulfur atom, oxygen atom and the NH group on the transfer of the electronic effect of substituents in the studied systems is discussed. It was found that the substituent effects are transferred better in 3-arylmethylenephthalides than in 3-arylmethylenethiophthalides and 3-arylmethylenephthalimidines. The formerly expressed supposition, that in 3-arylmethylenephthalides the transfer of the electronic effect of the substituents takes place mainly *via* *p*-electrons of the lactone oxygen atom, was confirmed. The preparation of some new derivatives of 3-phenylmethylenephthalimidine and 3-phenylmethylenethiophthalide was also described.

In preceding papers<sup>1,2</sup> we studied the transfers of the electronic effects of substituents in 3-arylmethylenephthalides or 3-aryloxymethylenephthalides and 3-arylthiomethylenephthalides by means of linear relationships between the C=O stretching frequencies and the  $\sigma$ -constant of substituents. The IR spectra of phthalimidine and some its derivatives, measured in chloroform or in dioxan, were published in papers<sup>3,4</sup>. The spectrum of unsubstituted 3-phenylmethylenethiophthalide was interpreted in paper<sup>5</sup>. The IR spectra of 3-arylmethylenephthalimidines and 3-arylmethylenethiophthalides have not yet been systematically studied.

In this paper, in continuation of our preceding papers<sup>1,2</sup>, we deal with the study of the transfer of the electronic effect of substituents. The study is carried out by investigating the C=O stretching vibration region of the IR spectra of substituted *trans*-3-phenylmethylenephthalimidines (I) and *trans*-3-phenylmethylenethiophthalides (II).

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## EXPERIMENTAL

### Compounds Studied

*trans*-3-Arylmethylenephthalimidines *I* were prepared by Gabriel modification of Perkin condensation of phthalimide with corresponding arylacetic acids. In comparison with earlier methods of preparation of 3-phenylmethylenephthalimidine<sup>6,7</sup> the method mentioned gives relatively good yields of 3-arylmethylenephthalimidines, *i.e.* 40–70%. The condensation requires 2–3 hours and 260–270°C for completion if equimolar amounts of phthalimide and corresponding arylacetic acid are used in the presence of potassium acetate. After neutralisation of the reaction mixture the separated crude product was freed from phthalimide by repeated washing with hot water and crystallisation from ethanol. 3-(4-Aminophenylmethylene)phthalimidine was prepared by reduction of 3-(4-nitrophenylmethylene)phthalimidine with zinc in acetic acid and crystallisation from ethanol. As the studied 3-arylmethylenephthalimidines, with the exception of 3-phenylmethylenephthalimidine<sup>6,7</sup>, were not yet described in the literature, we list their melting points and elemental analyses in Table I.

*trans*-3-Arylmethylenethiophthalides *II* were prepared similarly as 3-arylmethylenephthalimidines, *i.e.* by condensation of thiophthalic anhydride with corresponding arylacetic acids. This method is different from that described in the literature<sup>5</sup>. The yields of substituted 3-phenylmethylenethiophthalides vary in the 25–38% interval. For condensation equimolar amounts of thiophthalic anhydride and the corresponding arylacetic acid were used in the presence of anhydrous potassium acetate. The reaction was completed after two hours at 225–235°C. The crude product was crystallised from ethanol. Because very low yields of 3-(4-nitrophenylmethylene)thiophthalide were obtained on condensation of thiophthalide with 4-nitrophenylacetic acid by the described method, we also carried out this reaction in acetic anhydride at 120–125°C, the reaction time being 6 hours. The reaction mixture obtained was diluted with water and the separated crude product was recrystallised from ethanol–chloroform (1:1). As the studied 3-arylmethylenethiophthalides, with the exception of 3-phenylmethylenethiophthalide<sup>5</sup>, have not yet been described, we present their melting points and elemental analyses in Table II. The preparation, melting point, and analytical data of 3-(4-aminophenylmethylene)thiophthalide<sup>8</sup> will be described in a subsequent paper.

All compounds were purified by crystallisation before spectral measurements.

### Spectral Measurements and Treatment of Data

The infrared spectra were measured on a Zeiss UR 20 spectrophotometer provided with a prism monochromator. For 3-arylmethylenephthalimidines (*I*) the regions 1800–1600  $\text{cm}^{-1}$  and 3500–3400  $\text{cm}^{-1}$  were chosen, while 3-arylmethylenethiophthalides (*II*) were measured in the 1800–1600  $\text{cm}^{-1}$  region. The frequency scale of the spectrophotometer was calibrated according to Jones and coworkers<sup>9</sup> using a standard mixture of indene, cyclohexanone, and camphor. For measurements  $8 \cdot 10^{-3}$  molar solutions of compounds in chloroform and NaCl cells of 0.1 cm

TABLE I  
Melting Points and Analytical Data of New Derivatives of *trans*-3-Phenylmethylenephthalimides I

X	M.p., °C	Formula (M.w.)	Calculated/Found		
			% C	% H	% N
4-NH <sub>2</sub>	199—200 decomp.	C <sub>15</sub> H <sub>12</sub> N <sub>2</sub> O (236·3)	76·25	5·12	11·85
			76·31	5·03	12·06
4-OCH <sub>3</sub>	202—203	C <sub>16</sub> H <sub>13</sub> NO <sub>2</sub> (251·2)	76·49	5·18	5·57
			76·61	5·36	5·49
3-CH <sub>3</sub>	165—166	C <sub>16</sub> H <sub>13</sub> NO (235·2)	81·70	5·53	5·95
			81·48	5·31	6·09
4-F	231—232	C <sub>15</sub> H <sub>10</sub> FNO (239·2)	75·31	4·21	5·85
			75·44	4·26	5·61
3-OCH <sub>3</sub>	144—146	C <sub>16</sub> H <sub>13</sub> NO <sub>2</sub> (251·2)	76·49	5·18	5·57
			76·22	4·74	5·72
4-I	232—234	C <sub>15</sub> H <sub>10</sub> INO (347·2)	51·88	2·90	4·03
			51·76	2·63	4·14
4-Cl	236	C <sub>15</sub> H <sub>10</sub> ClNO (255·7)	70·45	3·94	5·47
			70·22	3·87	5·66
4-Br	233	C <sub>15</sub> H <sub>10</sub> BrNO (300·2)	60·01	3·35	4·69
			60·26	3·41	4·46
3-F	205—206	C <sub>15</sub> H <sub>10</sub> FNO (239·2)	75·31	4·21	5·85
			75·12	4·08	5·92
3-I	208	C <sub>15</sub> H <sub>10</sub> INO (347·2)	51·88	2·90	4·03
			51·62	2·74	4·24
4-NO <sub>2</sub>	241—242	C <sub>15</sub> H <sub>10</sub> N <sub>2</sub> O <sub>3</sub> (266·2)	67·66	3·78	10·52
			67·54	3·46	10·77

thickness were used. The absorption bands were measured with a  $\pm 1 \text{ cm}^{-1}$  accuracy. For the preparation of solutions chloroform of analytical grade was used, which was purified and dried in the usual manner.

When linear correlations of the spectral data with the structure of the compounds were studied,  $\sigma$ -constants of substituents, published by McDaniel and Brown<sup>10</sup>, were used. The parameters

TABLE II  
Melting Points and Analytical Data of New Derivatives of *trans*-3-Phenylmethylenethiophthalides II

X	M.p., °C	Formula (M.w.)	Calculated/Found		
			% C	% H	% S
4-OCH <sub>3</sub>	128–130	C <sub>16</sub> H <sub>12</sub> O <sub>2</sub> S (268.3)	71.65	5.04	11.95
			71.85	4.93	12.32
4-F	141–143	C <sub>15</sub> H <sub>9</sub> FOS (256.2)	70.32	3.52	12.49
			70.62	3.26	12.31
3-OCH <sub>3</sub>	97–99	C <sub>16</sub> H <sub>12</sub> O <sub>2</sub> S (268.3)	71.65	5.04	11.95
			71.83	5.24	12.18
4-I	168–170	C <sub>15</sub> H <sub>9</sub> IOS (364.1)	49.45	2.48	11.05
			49.32	2.68	11.48
4-Cl	116–118	C <sub>15</sub> H <sub>9</sub> ClOS (272.9)	64.48	3.52	11.98
			64.86	3.84	12.36
3-F	124–126	C <sub>15</sub> H <sub>9</sub> FOS (256.2)	70.32	3.52	12.49
			69.95	3.68	12.05
3-Cl	128–130	C <sub>15</sub> H <sub>9</sub> ClOS (272.9)	64.48	3.52	11.98
			64.63	3.61	11.61
4-NO <sub>2</sub>	240–242	C <sub>15</sub> H <sub>9</sub> NO <sub>3</sub> S (283.4)	63.40	3.17	11.28
			63.11	2.98	11.46

of linear correlations were computed using an automatic digital computer Gier and statistical relationships published in the literature<sup>11</sup>. The measured values of the frequencies and  $\sigma$ -constants are listed in Tables III and IV. The linear dependences of C=O stretching frequencies on  $\sigma$ -constants of substituents are presented in Figs 1 and 2.

## RESULTS AND DISCUSSION

In the IR spectra of 3-arylmethylenephthalimidines I a strong absorption band belonging to the C=O stretching vibration (Table III) is observed in the 1718 to 1705 cm<sup>-1</sup> region. In the case of 3-arylmethylenethiophthalides II the frequencies of the C=O stretching bands are shifted to lower values, *i.e.* 1697–1685 cm<sup>-1</sup> (Table IV). We observed already in our previous works<sup>1,2</sup> that 3-arylmethylenephthalides, 3-aryloxymethylenephthalides, and 3-arylythiomethylenephthalides display

TABLE III  
Spectral Data and  $\sigma$ -Constants of Substituents for Substituted *trans*-3-Phenylmethylenephthalimides I

Compound No	X	$\sigma$	$\nu(\text{C}=\text{O})$ $\text{cm}^{-1}$	$\nu(\text{C}=\text{C})$ $\text{cm}^{-1}$	$\nu(\text{N}-\text{H})$ $\text{cm}^{-1}$
1	4-NH <sub>2</sub>	-0.66	1 705	1 660	—
2	4-OCH <sub>3</sub>	-0.27	1 708	1 660	3 447
3	3-CH <sub>3</sub>	-0.07	1 710	1 659	3 446
4	H	0.00	1 711	1 660	3 447
5	4-F	0.06	1 711	1 668	3 447
6	3-OCH <sub>3</sub>	0.12	1 712	1 661	3 445
7	4-I	0.18	1 712	1 659	3 447
8	4-Cl	0.23	1 713	1 663	3 447
9	4-Br	0.23	1 713	1 660	3 447
10	3-F	0.34	1 714	1 668	3 446
11	3-I	0.35	1 713	1 663	3 445
12	4-NO <sub>2</sub>	0.78	1 718	1 660	3 446

TABLE IV  
Spectral Data and  $\sigma$ -Constants of Substituents for Substituted *trans*-3-Phenylmethylenethiophthalides II

Compound No	X	$\sigma$	$\nu(\text{C}=\text{O})$ $\text{cm}^{-1}$	$\nu(\text{C}=\text{C})$ $\text{cm}^{-1}$
13	4-NH <sub>2</sub>	-0.66	1 685	1 643
14	4-OCH <sub>3</sub>	-0.27	1 688	1 642
15	H	0.00	1 690	1 642
16	4-F	0.06	1 691	1 643
17	3-OCH <sub>3</sub>	0.12	1 691	1 638
18	4-I	0.18	1 693	1 640
19	4-Cl	0.23	1 693	1 642
20	3-F	0.34	1 694	1 640
21	3-Cl	0.37	1 694	1 643
22	4-NO <sub>2</sub>	0.78	1 697	1 643

complex C=O stretching bands. The complex character of these absorption bands is in most cases deducible from the high value of the band half-width ( $\Delta\nu_{1/2} = 30-40 \text{ cm}^{-1}$ ). Primary maxima, the intensity ratio of which is close to unit, are practically inaccessible without mathematical analysis. Similar to the mentioned compounds, 3-arylmethylenephthalimidines *I* and 3-arylmethylenethiophthalides *II* give broad C=O stretching bands ( $\Delta\nu_{1/2} = 30-35 \text{ cm}^{-1}$ ) which may also be considered as complex. The values given in Tables III and IV, similar to those published in previous papers<sup>1,2</sup>, represent the frequencies of the complex C=O stretching bands. Although the formation of the complex band of the C=O stretching vibration of the mentioned compounds remains as yet unexplained, it can be surmised that the cause

of this effect should be looked for in the  $\begin{array}{c} \text{O} \\ \parallel \\ -\text{C}-\text{Z}-\text{C}=\text{CH}- \\ | \end{array}$  grouping, (where Z = O, NH, S) which is present in all compounds studied. This supposition is also supported by the fact that when the reduction of 3-arylmethylenephthalides leads

to the reduction of the C=C bond in the  $\begin{array}{c} \text{O} \\ \parallel \\ -\text{C}-\text{O}-\text{C}=\text{CH}- \\ | \end{array}$  grouping, a simple C=O stretching band<sup>12</sup> appears in the spectrum. With phthalide itself, in which

the  $\begin{array}{c} \text{O} \\ \parallel \\ -\text{C}-\text{Z}-\text{C}=\text{CH}- \\ | \end{array}$  grouping is absent, an unsplit band has also been observed in the region of the C=O stretching frequencies<sup>13</sup>. Supposing the mentioned facts and the fact that the maxima of the components of the complex band have in all instances the ratio of intensities close to unit, we can state that the frequencies of the complex C=O bands of compounds *I* and *II* (Tables III and IV) may be compared with the frequencies of the complex C=O bands published in earlier papers<sup>1,2</sup>, without being afraid of committing any great inaccuracy.

The C=O stretching frequencies of 3-arylmethylenephthalimidines *I* and 3-arylmethylenethiophthalides *II* can be well correlated with the  $\sigma$ -constants of substituents (Tables III and IV, Figs. 1, 2). Statistical parameters of these correlations are given in Table V. For comparison we have also included into this Table the parameters of linear correlations  $\nu(\text{C}=\text{O})-\sigma$  for the series of 3-arylmethylenephthalides (*III*), 3-aroxyethylenephthalides (*V*) and 3-arylthiomethylenephthalides (*IV*) described in our previous papers<sup>1,2</sup>.

From the comparison of the slopes of the  $\nu(\text{C}=\text{O})-\sigma$  relations for compounds of the *I-III* type it follows that electronic effects are transferred better in 3-arylmethylenephthalides *III* than in 3-arylmethylenethiophthalides *II* and 3-arylmethylenephthalimidines *I*. In the latter two cases the transfer is approximately equal. In order to be able to discuss the given problem more thoroughly we must first elucidate the question by which route the electronic effects of substituents will be transferred in the systems under study. Essentially, for substances of the *I-V* type (Table V), two pos-

sible routes of the transfer of the electronic effect of the substituent onto the carbonyl group may be supposed. The first possibility is that the transfer takes place *via* the benzene ring on which the carbonyl group is bound. The second type of transfer can take place *via* the atom or group Z. In our previous paper<sup>1</sup> we expressed the supposition that in 3-arylmethylenephthalides *III* the effects of substituents are transferred mainly *via* the oxygen atom ( $Z=O$ ) of the lactone ring. This supposition was based on the comparison of the slopes of the  $\nu(C=O)-\sigma$  relations for 3-arylmethylenephthalides *III* and for their heterocyclic analogue, 3-arylmethylenecinchomeronides. The practical identity of the values of the slopes for both types of compounds confirmed that the nitrogen heteroatom in the aromatic ring does not practically influence the transfer of the substituent effect, and therefore, the latter must take place by a different route, than through the aromatic ring. On the other hand, when comparing the values of the slopes of the  $\nu(C=O)-\sigma$  relations for the compounds of *I-III* type it can be observed that the substitution of  $Z=O$  by  $Z=S$  or  $NH$  changes the value of the slopes from 12.02 to 8.75 or 8.99. From this it follows that the atom or group Z distinctly influences the transfer of the electronic effect of the substituent onto the carbonyl group. This confirms our supposition<sup>1</sup> that in the case of 3-arylmethylenephthalides *III* the transfer takes place mainly *via* the *p*-electrons of the lactone oxygen. The question is whether in 3-arylmethylenethio-

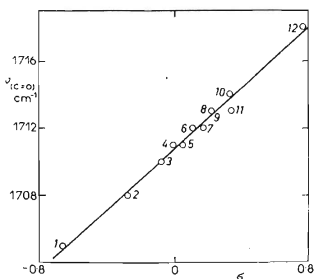


FIG. 1

Dependence of the Frequencies of the C=O Stretching Vibration on  $\sigma$ -Constants of Substituents for *trans*-3-Arylmethylenephthalimidines *I*

The numbering of the compounds is the same as in Table III.

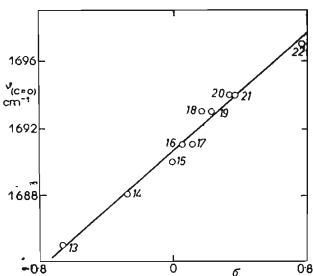


FIG. 2

Dependence of the Frequencies of the C=O Stretching Vibration on  $\sigma$ -Constants of Substituents for *trans*-3-Arylmethylenethiophthalides *II*

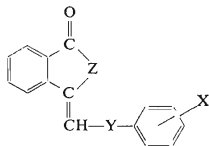
The numbering of the compounds is the same as in Table IV.



phthalides *II* and 3-arylmethylenephthalimidines *I* the electronic effects are also transferred *via* the sulfur atom of the NH group. In Table V it can be seen that the values of the slopes of the  $\nu(\text{C}=\text{O})-\sigma$  relations for the compounds of type *I* and *II* are practically identical and that it is therefore difficult to answer this question unequivocally. In our previous paper<sup>2</sup> we observed on the basis of the comparison of the slopes of the  $\nu(\text{C}=\text{O})-\sigma$  relations that the effects of the substituents are better transferred *via* the oxygen atom in 3-aryloxymethylenephthalides *V* than through the sulfur atom in 3-arylthiomethylenephthalides *IV*. When expressing the ratio of the slopes for the mentioned compounds by a coefficient, the following value is obtained:  $\eta_1 = \rho(\text{IV})/\rho(\text{V}) = 0.86$ . If the same coefficient is calculated for 3-arylmethylenethiophthalides *II* and 3-arylmethylenephthalides (*III*) the value  $\eta_2 = \rho(\text{II})/\rho(\text{III}) = 0.73$  is obtained which is comparable with the preceding coefficient  $\eta_1$ . From this it follows that probably also in 3-arylmethylenethiophthalides *II* the electronic effects of substituents are transferred *via* the sulfur atom in the thiolactone ring. Finally, if we mainly suppose that in 3-arylmethylenephthalimidines *I* the transfer is also brought about *via* the NH group, then the sequence of the magnitudes of the values

TABLE V

Statistical Parameters of Linear Correlations of the C=O Stretching Frequencies with  $\sigma$ -Constants of Substituents for Compounds of the Following Structure



*n* Number of compounds used for correlation;  $\rho$  slope of the regression line;  $s_\rho$  error of the slope;  $q$  intercept;  $s_q$  error of the intercept;  $s$  standard deviation;  $r$  correlation coefficient. All results were obtained from the data measured in chloroform solutions.

Type of compound	Z	Y	<i>n</i>	$\rho$	$s_\rho$	$q$	$s_q$	$s$	$r$
<i>I</i> <sup>a</sup>	NH	—	12	8.99	0.32	1 710.7	0.1	0.37	0.994
<i>II</i> <sup>a</sup>	S	—	10	8.74	0.44	1 690.6	0.2	0.51	0.990
<i>III</i> <sup>b</sup>	O	—	12	12.02	0.66	1 782.2	0.2	0.80	0.985
<i>IV</i> <sup>b</sup>	O	S	10	7.45	0.54	1 787.0	0.2	0.70	0.979
<i>V</i> <sup>b</sup>	O	O	13	8.68	0.50	1 786.9	0.1	0.60	0.982

<sup>a</sup> Results published in this paper. <sup>b</sup> Results published in preceding papers<sup>1,2</sup>.



of the computed slopes of  $\nu(\text{C}=\text{O})-\sigma$  relations —  $\varrho(\text{III}) > \varrho(\text{II}) \cong \varrho(\text{I})$  — is in agreement with the results from the paper by Marcus and coworkers<sup>14</sup>; moreover, the atom or group  $Z$  in the compounds of the type  $I-III$  acts as an electron donor in the conjugated system.

When comparing the absolute members of the regression equations ( $q$ ) in Table V, *i.e.* the calculated  $\text{C}=\text{O}$  stretching frequencies for unsubstituted derivatives in series of compounds of the type  $I-III$ , we can observe the following sequence of the magnitudes of  $q$ -values:  $q(\text{III}) \gg q(\text{I}) > q(\text{II})$ . The difference between the  $\text{C}=\text{O}$  stretching frequencies of 3-arylmethylenephthalides  $III$  and of 3-arylmethylenephthalimidines  $I$ , which is approximately  $70 \text{ cm}^{-1}$ , is comparable to the difference between the  $\text{C}=\text{O}$  stretching frequencies of acyclic esters and amides of carboxylic acids<sup>15</sup>. However, when comparing the frequencies of 3-arylmethylenephthalides  $III$  with the  $\text{C}=\text{O}$  frequencies of 3-arylmethylenethiophthalides  $I$ , it can be observed that the difference, which is approximately  $90 \text{ cm}^{-1}$ , is much higher than that usually found between the  $\text{C}=\text{O}$  frequencies of acyclic esters and thioesters of carboxylic acids<sup>16,17</sup>. From this it follows that the bulk of the sulfur atom in the five-membered ring plays an important role. When comparing the covalent radius of the sulfur atom ( $1.04 \text{ \AA}$ ) with the covalent radii of oxygen ( $0.66 \text{ \AA}$ ) and nitrogen atoms ( $0.70 \text{ \AA}$ ), we can observe that if the strain in the five-membered ring of the compounds of type  $I$  and  $III$  is approximately equal, then the strain in the five-membered ring in compounds of type  $II$ , containing a sulfur atom, should be much weaker. The mentioned fact causes the  $\text{C}=\text{O}$  stretching frequencies of 3-arylmethylenethiophthalides  $II$  to be shifted to lower values with respect to those of their oxygen analogues to a much greater extent than the  $\text{C}=\text{O}$  frequencies of acyclic thioesters of carboxylic acids with respect to the corresponding esters. The  $\text{C}=\text{O}$  stretching frequencies of 3-arylmethylenethiophthalides  $II$  are already close to the frequencies of their acyclic analogues, which indicates a minimal strain in the five-membered thiolactone ring.

In the studied 3-arylmethylenephthalimidines a band of low to medium intensity is observed in the  $1668-1659 \text{ cm}^{-1}$  region, belonging to the  $\text{C}=\text{C}$  stretching vibration of the central  $>\text{C}=\text{CH}-$  group. 3-Arylmethylenethiophthalides  $II$  display the same band in the  $1643-1638 \text{ cm}^{-1}$  region. In both types of compounds the positions of these bands are little sensitive to the solvent and substituent effects. When comparing the frequencies of the  $\text{C}=\text{C}$  stretching vibration in compounds of type  $I$  and  $II$  with those of the  $\text{C}=\text{C}$  stretching vibrations of 3-arylmethylenephthalides<sup>1</sup> ( $1671-1668 \text{ cm}^{-1}$ ) it can be observed that the sequence of the magnitudes is the same as in the case of the  $\text{C}=\text{O}$  stretching frequencies, *i.e.*  $\nu(\text{III}) > \nu(\text{I}) > \nu(\text{II})$ .

In the spectra of 3-arylmethylenephthalimidines  $I$  ( $3447-3445 \text{ cm}^{-1}$  region) a weak absorption band belonging to the  $\text{N}-\text{H}$  stretching vibration is observed. Similarly, as well as the absorption bands of the  $\text{C}=\text{C}$  stretching vibration, these bands are also little sensitive to the effects of substituents.

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