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^{17}O NMR Study of Steric Interactions in Hindered N-Substituted Imides

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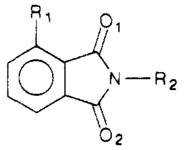
^{17}O NMR spectroscopic data (natural abundance in acetonitrile at 75 °C) were obtained for a series of N-substituted phthalmides (1–11), a series of N-substituted succinimides and maleimides (12–19), and N-substituted phthalamides (20–22). The ^{17}O NMR data showed large deshielding effects as the steric bulk of the N-substituent increased; introduction of a 3-substituent in the phthalimide series produced additional deshielding. The deshielding effects of 3-substitution and N-substitution in the phthalimides were found to be roughly additive. The results correlated with in-plane bond angle distortions (X-ray results and molecular mechanics calculations). The results for *N*-arylphthalimides showed that the aromatic ring was rotated out of the plane (torsion angle effect) in those cases.

^{17}O NMR spectroscopy is rapidly developing into an important method for examining a wide variety of structural problems[1] and may provide new insights into the understanding of chemical reactivity.[2] A number of ^{17}O NMR studies have focused on electronic effects,[3] while others have investigated conformational effects.[4] Recent work has shown that quantitative relationships can be formulated between ^{17}O NMR data and torsion angles for aromatic nitro compounds,[5] acetophenones,[6] aryl ketones,[7] and aromatic carboxylic acids and derivatives.[8] Thus structural information relating to torsion angle relationships is accessible readily by ^{17}O NMR methodology. ^{17}O studies on anhydrides have shown sensitivity to structural variations.[9] Recently, ^{17}O NMR data[2] on a series of hindered 3-substituted phthalic anhydrides and corresponding phthalides have been shown to correlate with in-plane bond angle distortions. The nontorsional ^{17}O chemical shift effects observed for the anhydrides, thought to be indicative of repulsive van der Waals interactions, could be used[2] to explain the regiospecificity of reduction reactions. We report here ^{17}O NMR data for a series of N-substituted phthalimides, N-substituted phthalamides, and a series of N-substituted succinimides and maleimides which show that the ^{17}O NMR chemical shift data provide new insights into structure and reactivity in relation to steric phenomena.

Table I. ^{17}O Chemical Shift Data[a] for Substituted Phthalimides in CH_3CN at 75 °C



compd	R_1	R_2	$\delta(C{=}O)_1$	$\delta(C{=}O)_2$	δ(R)
1	H	H	379.0	379.0	
2	H	Me	374.0	374.0	
3	H	*i*-Pr	383.0	383.0	
4	H	*t*-Bu	394.0	394.0	
5	*t*-Bu	H	407.3	370.6	
6	*t*-Bu	*t*-Bu	423.3	385.3	
7	H	Ph	378.3	378.3	
8	H	2-MeC_6H_4	381.3	381.3	
9	H	2,6-$Me_2C_6H_3$	384.0	384.0	
10	H	4-$MeOC_6H_4$	377.5	377.5	50.7
11	H	2-Me-4-$MeOC_6H_3$	380.0	380.0	51.0

[a] Natural abundance with 2-butanone internal standard; data point resolution ±0.2 ppm; reproducibility ≤±1 ppm.

Results

^{17}O NMR data were obtained (natural abundance) for a series of N-substituted phthalimides (1–11), a series of

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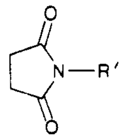
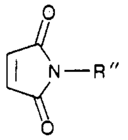
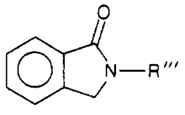
Table II. ^{17}O NMR Chemical Shift Data (±1 ppm)[a] for Selected Imides and Phthalamides in Acetonitrile at 75 °C



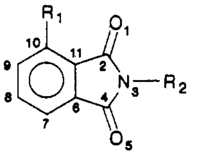




compd	R′	δ(C=O)	compd	R″	δ(C=O)	compd	R‴	δ(C=O)
12	H	373.5	**16**	H	411	**20**	H	282
13	Me	371	**17**	Me	407	**21**	Me	281
14	*t*-Bu	392	**18**	*t*-Bu	426	**22**	*t*-Bu	300
15	Ph	376	**19**	Ph	412			

[a] Natural abundance with 2-butanone or acetone as internal standard.

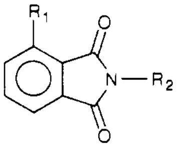
Table III. Molecular Mechanics Calculated Bond Angles (deg, ±1°) and X-ray Data for Phthalimides 1, 4, and 6



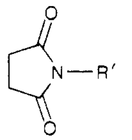
		1		**4**		**6**	
entry	angle	MM2	X-ray[a]	MM2	X-ray[b]	MM2	X-ray[c]
1	$O_1C_2N_3$	124	124.8	126	126.3	124	125.5
2	$C_{11}C_2N_3$	105	105.2	107	108.6	108	106.8
3	$C_{10}C_{11}C_2$	131	130.0	131	131.9	133	132.8
4	$C_2N_3C_4$	114	112.2	111	110.6	111	110.5
5	$N_3C_4O_5$	124	125.4	126	129.6	126	126.6
6	$N_3C_4C_6$	105	106.2	107	104.6	107	107.4
7	$C_4C_6C_7$	131	120.3	131	125.9	128	127.2
8	$C_2N_3R_2$	122		122	122.1	122	122.6
9	$C_{11}C_{10}R_1$					126	125.2

[a] Literature values taken from ref 14. [b] σ values ~1.5. [c] σ values ~0.5.

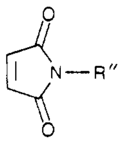
N-substituted succinimides (**12–15**) and maleimides (**16–19**), and several substituted phthalamides (**20–22**) in acetonitrile at 75 °C. The data for the phthalimides are summarized in Table I. With the exception of the parent compound **1**, the signal for the carbonyl oxygens was deshielded as the size of the N-substituent increased, despite similar electronic effects (shielding) of the alkyl groups. For example, the substitution of a *N*-isopropyl group for an *N*-methyl group yielded a 9 ppm downfield shift while the similar effect of the *N*-*tert*-butyl group was 20 ppm (compare **2–4**). In the unsymmetrical compound **5** separate signals for both carbonyl oxygens were detected. Since electronic effects are again negligible, the large difference (deshielding) observed was indicative of significant van der Waals interactions. the magnitude of this shift (28 ppm) suggested in-plane distortions caused by the relief of the steric interactions of the substituent on the aromatic ring with the carbonyl oxygen similar to that observed[2] in the analogous phthalic anhydride system (~25 ppm). The results for the doubly hindered compound **6** showed that the ring substituent deshielding effect and that due to the N-substituent were additive (vide infra).



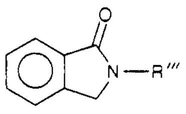
1-11



12-15



16-19



20-22

The data for *N*-arylphthalimides **7–11** were not consistent with expectations based on electronic effects. The effect of methyl substitution on the *N*-phenyl group would be expected to result in slight shielding of the ^{17}O signal. However, the data clearly showed that *o*-methyl substitution caused modest deshielding. For example, one *o*-methyl resulted in a 3 ppm deshielding effect, while two *o*-methyl groups caused a 6 ppm downfield shift (compare **7–9**). Thus the effects of *o*-methyl groups appear to be additive and suggestive of repulsive van der Waals interactions. In compounds **10–11** with *p*-methoxy groups, the electronic influence was again negligible. This result suggests that the substituted *N*-phenyl group is not coplanar with the imide ring system. The chemical shift value for compound **11** also showed that the *o*-methyl group caused roughly a 3 ppm deshielding effect consistent with those for **8** and **9**.

The large deshielding effects noted in **2–4** were surprising. The ^{17}O chemical shift data for N-substituted succinimides **12–15** and maleimides **16–19** were examined to test the generality of this finding (Table II). The ^{17}O NMR chemical shift data for the N-substituted succinimides and maleimides showed deshielding effects identical with those for the phthalimides. In addition ^{17}O NMR data for the analogous N-substituted phthalamides **20–22** showed deshielding effects with large N-substituents in agreement with the above three imide systems. The signals for compounds with *N*-*tert*-butyl groups were deshielded 20 ± 1 ppm relative to those for the *N*-Me compounds for all four cases. The signals for *N*-phenyl compounds were deshielded by 5 ± 1 ppm relative to those for the *N*-methyl compounds. The chemical shift data for the parent compounds (R = H) of each group (**1**, **12**, **16**, **20**) are complicated by the presence of a hydrogen-bonding component. Simple hydrogen bonding to a carbonyl group should clearly result in an upfield shift of the ^{17}O signal.[3a,10,11] The

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effect of N–H donation to another system on the carbonyl of the donor imide is not as clear.[11,12] The overall effect of differential hydrogen bonding is complex.[11] Thus the chemical shift differences between these compounds and those for the N-substituted compounds were difficult to interpret.

Discussion

Molecular mechanics (MM2) calculations[13] were carried out on **1**–**4**; see Table III for selected bond angles (entries 1–9). The calculations predicted in-plane angle distortions of the planar molecules with the larger N-substituents. The bond angle of the carbonyl group opened toward the ring (entries 1 and 2), and the internal bond angle of the imide (entry 4) diminished as the N-substituent increased in size. In contrast to the 3-substituted anhydride system, the distortion for the N-substituted imides resulted in a symmetrical opening of the carbonyl angles (entries 1 and 2, Table III). The X-ray structure of phthalimide **1** has been reported,[14] and we have obtained the X-ray structure of **4**.[15] The structures were found to remain planar in agreement with molecular mechanics calculations. Unfortunately, the X-ray results show that the crystal structure for **1** and **4** are not symmetrical around an axis through the nitrogen bisecting the molecule, making quantitative comparisons difficult. Despite this, the bond angle distortions noted were in qualitative agreement with those predicted by the calculations (Table III).

The ^{17}O signal for the double hindered carbonyl of **6** was deshielded by 50 ppm, which was much larger than any other effects seen. Moreover, the magnitude of the deshielding effect on the signal for the other carbonyl was consistent with expectations based upon the ^{17}O data for **1**, **4**, and **5**. The X-ray structure of **6** was obtained to compare with molecular mechanics calculations (Table III) and again confirmed the molecule to be planar. The calculations predicted essentially identical values for the angles for $(C{=}O)_2$ in both structures **4** and **6** and the X-ray results are qualitatively in agreement; compare entries 1 and 5 for compounds **4** and **6**. The doubly hindered carbonyl, $(C{=}O)_1$, is being influenced in opposing directions by the two *tert*-butyl groups such that no unusual distortion is apparent in the structure. However, the ^{17}O results show that this carbonyl, $(C{=}O)_1$, is subject to severe van der Waals interactions.

Previous dipole moment studies[16] on *N*-arylphthalimides had been interpreted to show coplanarity of the two aryl ring systems. IR studies found little variation in carbonyl absorption frequency with *N*-aryl substituent,[17] suggesting that the extent of conjugation between the *N*-aryl group and the carbonyl group was small. More recently an NMR study[18] on this system concluded that no consistent correlation with size of aryl ortho substituents or their expected electronic properties with carbonyl ^{13}C chemical shift values was evident. The ^{17}O NMR data for **7**–**11** showed little or no electronic influence; however, van der Waals interactions were clearly the predominant influence. Molecular mechanics calculations for **7**–**11** predicted dihedral angles (torsion angles) of the aryl ring with the phthalimide system of 50°, 64°, 75°, 50°, and 65°, respectively. The X-ray structure of **7** showed a dihedral angle of 56° in reasonable agreement with the calculations. A plot of ^{17}O δ vs. calculated dihedral angle for **7**–**9** is shown in Figure 1. A reasonable correlation was found which suggested that the ^{17}O NMR data for the *N*-arylphthalimides resulted from the minimization of van der Waals repulsions by torsion angle rotation (loss of overlap) of the *N*-aryl group. Interestingly, since the ^{17}O data still shows deshielding effects, minimization of van der Waals repulsion is not complete. This suggests that some conjugation between the two systems is retained at the expense of complete minimization of van der Waals interactions. The ^{17}O results are consistent with the IR and ^{13}C NMR results and provide new perspectives into the competing influences in this system.

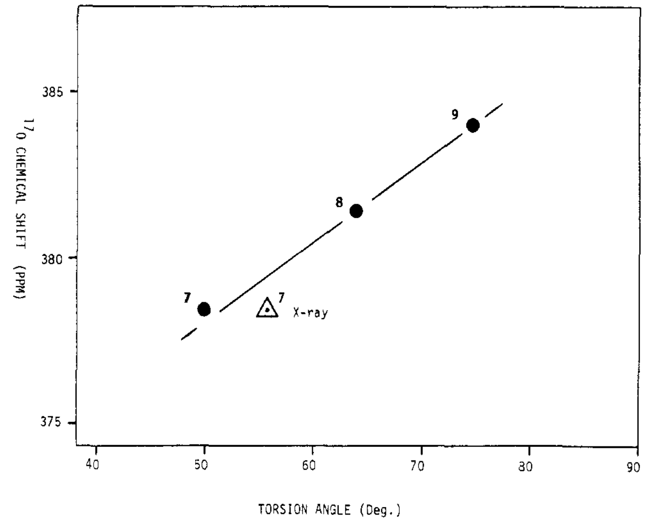

Figure 1. Plot of ^{17}O NMR chemical shift data for *N*-arylphthalimides **7**–**9** vs. calculated (MM2) torsion angles.

The ^{17}O NMR results reported here give interesting new insights into ground-state structure. In certain systems (cf. **6** or **7**–**11**) the ^{17}O method provides detailed information not accessible by other methods. The results for planar amides (**20**–**22**) show that the N-substituent deshielding effects are not limited to imides. However, it is not clear that these effects will be predominant in conformationally mobile (acyclic) systems. The ^{17}O NMR results may provide insights into the regiospecificity of imide reductions. For reductions in which electron transfers are rate determining, one would expect the carbonyl which shows the most deshielded ^{17}O chemical shift value to undergo reaction preferentially. For example, the regiospecific zinc reduction of a hindered imide[19] is consistent with the above expectation. Thus ^{17}O NMR continues to show promise as a means to probe organic structure and reaction mechanisms.

Experimental Section

Molecular mechanics calculations were carried out by use of the program available from Professor C. Still, Columbia University. X-ray analysis (**4**, **6**, **7**) were obtained by Dr. D. VanDerveer at the Georgia Institute of Technology. Mass spectra were obtained

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(15) The crystal for compound **4** proved to be meta stable under analysis conditions; thus errors reported in Table III are slightly larger than normal. A complete listing of X-ray structural data for **4**, **6**, and **7** will be provided upon request.

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on an instrument supported in part by NSF at Georgia Tech. Analyses were performed at Atlantic Microlabs, Atlanta, GA. All compounds used in this study which were not commercially available were prepared and purified by the standard literature procedures. Compounds **1**, **12**, **16**, and **19** were commercially available (Aldrich). Compounds **5** and **6** are new and their preparation is described below. The remaining previously reported compounds were prepared by standard methods and their physical data were in accord with the literature values: **2**,[20] **3**,[21] **4**,[22] **7** and **8**,[23] **9**,[24] **10**,[25] **11**,[26] **13**,[27] **14** and **15**,[28] **17**,[29] **18**,[30] **20**,[31] **21** and **22**.[32] They were characterized by spectroscopic (^{1}H and ^{13}C NMR spectroscopy) and physical data. The synthesis of **5** and **6** required 3-*tert*-butylphthalic anhydride, the detailed synthesis[2] of which has not previously been reported.

3-*tert*-Butylphthalic Anhydride. A mixture of 5,5-dimethyl-1,3-hexadiene (cis/trans; 3 g : 27.2 mmol) and maleic anhydride (1.89 g = 19.2 mmol) in 10 mL of dry benzene was heated in a Parr bomb at 120 °C for 12 h. After removal of the solvent, the residue was crystallized from ligroin to give 3.8 g of a white crystalline product (yield, 95.2%; mp 120–130 °C): ^{1}H NMR ($CDCl_3$) 1.13 (s, 9 H), 3.50 (m, 5 H), 6.06 ppm (m, 2 H); IR (KBr) 1830, 1765 cm^{-1}. The adduct (a mixture of isomers, 3.2 g = 15.4 mmol) was aromatized by stirring in 10 mL of decalin under reflux conditions in the presence of 320 mg of Pd/C for 12 h. After removal of decalin at 40 °C (1.5 mm), the residue was crystallized from methanol (yield, 2.8 g, 87.5%; mp 108 °C): ^{1}H NMR ($CDCl_3$) 1.59 (s, 9 H), 7.86 ppm (m, 3 H); IR (KBr) 1760, 1840 cm^{-1}; ^{13}C NMR ($CDCl_3$) 163.3, 162.7, 154.2, 136.0, 133.6, 133.4, 128.1, 123.4, 35.7, 29.5 ppm. The X-ray structure is given in ref 2.

3-*tert*-Butylphthalimide (5). A mixture of 460 mg (2.25 mmol) of 3-*tert*-butylphthalic anhydride and 150 mg (2.50 mmol) of urea was heated at 135 °C for 20 min. The contents were melted and then re-fused. The phthalimide was extracted with ether. The ether layer was washed with water and dried over Na_2SO_4. After removal of ether, the crude product was crystallized from methanol: yield, 350 mg (77%); white crystals; mp 159–161 °C; ^{1}H NMR ($CDCl_3$) 1.50 (s, 9 H), 7.63 ppm (m, 3 H); IR (KBr) 1760, 1840 cm^{-1}; ^{13}C NMR ($CDCl_3$) 168.5, 168.0, 152.0, 134.9, 133.9, 131.9, 129.1, 121.2, 35.7, 30.0 ppm. Anal. C, H.

***N*,3-Di-*tert*-butylphthalimide (6).** A mixture of 2.6 g (12.7 mmol) of *tert*-butylphthalic anhydride (66.7 mmol), *tert*-butylamine (distilled from KOH), and 7 mL of glacial acetic acid (distilled from P_2O_5) was heated under reflux conditions under nitrogen for 2 h. After being poured into ice–water, the phthalimide was extracted with CH_2Cl_2. The CH_2Cl_2 layer was washed with water and dried over Na_2SO_4. Removal of the solvent left a solid residue, which was recrystallized from methanol to yield 2.05 g (64%) of the phthalimide: mp 90 °C; ^{1}H NMR 1.50 (s, Ar, 9 H), 1.77 (s, 9 H), 7.56 ppm (m, 3 H); IR (KBr) 1700, 1760 cm^{-1}; ^{13}C NMR ($CDCl_3$) 169.6, 169.0, 150.7, 134.6, 133.2, 131.3, 128.6, 120.4, 57.8, 35.6, 30.1, 29.4 ppm. Anal. C, H.

The ^{17}O spectra were recorded on a JEOL GX-270 or on a Varian VXR-400 spectrometer equipped with a 10-mm broadband probe. All spectra were acquired at natural abundance at 75 °C in acetonitrile (Aldrich, anhydrous gold label under nitrogen) containing 1% of 2-butanone or acetone as an internal standard. The concentration of the carbonyl compounds employed in these experiments was 0.5 M. The signals were referenced to external deionized water at 75 °C. The 2-butanone resonance (558 ± 1 ppm) or acetone (571 ± 1 ppm) was used as an internal check on the chemical shift measurements for these compounds. The instrumental settings for the GX-270 at 36.5 MHz were spectral width 25 kHz, 2K data points, 90° pulse angle (28-μs pulse width), 200-μs acquisition delay, 40-ms acquisition time, and 40 000–100 000 scans. The instrumental settings for the VXR-400 at 54.22 MHz were spectral width 35 kHz 2K data points, 90° pulse angle (40-μs pulse width), 200-μs acquisition delay, 29-ms acquisition time, and ca. 20 000 scans. The spectra were recorded with sample spinning and without lock. The signal-to-noise ratio was improved by applying a 25-Hz exponential broadening factor to the FID prior to Fourier transformation. The data point resolution was improved to ±0.1 ppm on the VXR-400 and ±0.2 ppm on the GX-270 by zero filling to 8K data points. The reproducibility of the chemical shift data is estimated to be $\leq \pm 1.0$.

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