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ORIGINAL PAPER

Synthesis and Crystal Structure Studies of Three *N*-Phenylphthalimide Derivatives

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Abstract The three *N*-phenylphthalimide derivatives, 2-(3,4-dichlorophenyl)isoindoline-1,3-dione (**I**), 2-(2,4-dichlorophenyl)isoindoline-1,3-dione (**II**) and 2-(2,4,5-trichlorophenyl)isoindoline-1,3-dione (**III**), were synthesized by the condensation of equimolar amounts of phthalic anhydride and 3,4-dichloroaniline, 2,4-dichloroaniline, 2,4,5-trichloroaniline, respectively, under acetic acid reflux and their structures determined by a combination of elemental analysis, FT-IR, ^{1}H & ^{13}C-NMR spectroscopy and single crystal X-ray diffraction studies. Compounds **I** and **II** crystallize in a monoclinic crystal system (space group $P2_1/c$) with cell parameters of $a = 5.7414(2)$, $b = 8.0917(6)$, $c = 26.077(1)$ Å and $\beta = 99.4709(12)^{o}$ for compound **I**, and $a = 12.7133(9)$, $b = 13.4328(9)$, $c = 7.2603(5)$ Å and $\beta = 93.210\ (2)^{o}$ for compound **II**. On the other hand, compound III crystallizes in a tetragonal crystal system (space group $I4_1/a$) with $a = 13.4607(9)$ and $c = 30.100(2)$ Å. The phthalimide moieties of these compounds are essentially planar, while the chloro-substituted phenyl ring of each compound shows consistent twist from the phthalimide plane with dihedral angles of 61.02(3), 69.09(3) and 85.78(5)^{o}, respectively, for **I**, **II** and **III**. In the crystal structures of these compounds, a few weak C–H···O interactions form double-tape structures of centrosymmetric dimers of graph-set notation $R_2^2(10)$ for **I** and **III**, and an inversion dimer of graph-set motif $R_2^2(14)$ for **II**. In addition, some short contacts of C···C, C···O and Cl···Cl are observed for **I**, **II** and **III**, respectively.

Keywords *N*-Phenylphthalimide · Crystal structure · Intermolecular contact · Graph-set

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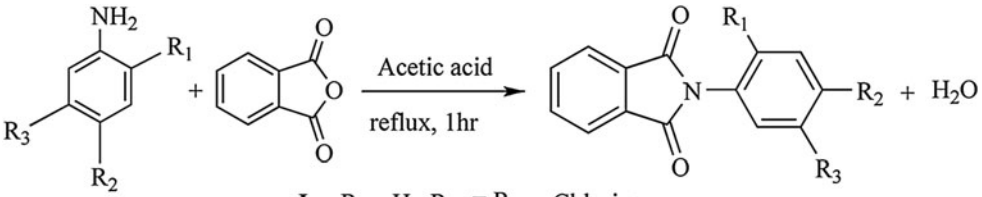
Introduction

The search for anti-cancer drugs has turned up many cytotoxic DNA binding and intercalating agents of diverse structure and function, several of which form the foundation of current chemotherapy. Phthalimides, *N*-substituted phthalimides, naphthalimides and bis-naphthalimides are a class of compounds with high antitumor activity on a variety of murine and human tumour cells [1]. These compounds have the ability to bind DNA by intercalation of the chromophores and two of them, mitonafide and amonafide, have been used in clinical trials. The therapeutic properties of these lead drugs were improved by designing bis-intercalating agents. One of these, elinafide, showed intense in vitro and in vivo activity and is currently being used in clinical trials against solid tumours [2]. It is thus reasonable to expect that members of this class of compounds will reach the oncology market in the near future. Phthalimides are well known cytotoxic DNA intercalating agents and have shown promise as potential anti-cancer agents. Its derivatives, such as bis-naphthalimides, represent a promising group of DNA-targeted anticancer agents, and the search for more potent analogues remains a priority [3–5]. A series of methylthiazonaphthalimides were synthesized and quantitatively

Scheme 1 The reaction scheme for the syntheses of compounds **I**, **II** and **III**



I; $R_1 = H$, $R_2 = R_3 =$ Chlorine

II; $R_1 = R_2 =$ Chlorine, $R_3 = H$

III; $R_1 = R_2 = R_3 =$ Chlorine

Table 1 Selected crystal data and experimental details of X-ray diffraction for compounds **I**, **II** and **III**

	I: $C_{14}H_7Cl_2NO_2$	**II**: $C_{14}H_7Cl_2NO_2$	**III**: $C_{14}H_6Cl_3NO_2$
Crystal data			
Formula weight	292.12	292.12	326.57
Crystal system	Monoclinic	Monoclinic	Tetragonal
Space group	$P2_1/c$	$P2_1/c$	$I4_1/a$
a (Å)	5.7414 (2)	12.7133 (9)	13.4607 (9)
b (Å)	8.0917 (6)	13.4328 (9)	–
c (Å)	26.077 (1)	7.2603 (5)	30.100 (2)
β (°)	99.4709 (12)	93.210 (2)	–
V (Å^3)	1194.96 (10)	1237.93 (15)	5453.8 (8)
Z	4	4	16
D (Mg m^{-3})	1.624	1.567	1.591
F000	592.00	592.00	2624.00
μ (mm^{-1})	0.54	0.52	0.67
Crystal size (mm)	0.34 × 0.20 × 0.14	0.37 × 0.28 × 0.15	0.44 × 0.29 × 0.26
Data collection			
θ_{max} (°)	30.0	30.0	30.0
θ_{min} (°)	3.2	3.0	3.0
h	−8→7	−17→17	−15→18
k	−11→11	−18→18	−16→18
l	−35→36	−10→10	−42→37
Measured reflections	21160	20941	22165
Independent reflections	3459	3592	3987
Cell parameters from reflections	18063	16725	14960
Reflections with $I > 2\sigma(I)$	3103	3122	2871
R_{int}	0.024	0.040	0.035
Refinement			
Refinement on	F^2	F^2	F^2
$R[F^2 > 2\sigma(F^2)]$	0.032	0.038	0.058
wR	0.088	0.104	0.179
S	1.05	1.07	1.08
Number of reflections	3459	3592	3987
Number of parameters	172	172	181
Weighting Scheme, w, $P = (Fo^2 + 2Fc^2)/3$	$1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.5569P]$	$1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.2431P]$	$1/[\sigma^2(F_o^2) + (0.0888P)^2 + 5.0189P]$
$(\Delta/\sigma)_{max}$	0.001	0.001	<0.001
$\Delta\rho_{min}$, $\Delta\rho_{max}$ (eÅ^{-3})	−0.40, 0.48	−0.25, 0.45	−0.70, 0.75

evaluated as efficient DNA intercalators, antitumor agents and DNA photocleavers [6]. The electronic structures of several *N*-substituted phthalimides have been investigated and reveal the presence of photofragmentation and photoelimination processes related to the decay of the aminium radical cation [7]. Naphthalimide derivatives are also gorgeous class of electron-deficient organic materials for OLEDs and are used as a new type of electron-transporting emitting materials for both small molecule and polymer-based OLEDs with high performance, good light stability, high fluorescent quantum yield, and high electron affinity [8, 9]. Phthalimide derivatives in the wake of the enormous biochemical and photochemical applicability have become important class of compounds for crystal structural studies. In a study of these class of compounds, it was observed that they are able to inhibit the acute inflammatory response, measured by inhibition of lipopolysaccharide (LPS)-induced TNF-α (Tumor necrosis factor-α) and neutrophil infiltration into mice lungs [10] with the phthalimide moiety demonstrated to be important for activity. Some study [11] also suggests phthalimide derivatives possess inhibitory effect on the HIV-1 reverse transcriptases. It is important hence for the crystal structures of these compounds to be studied since the structural and conformational features could affect the activity of these compounds. This study hence examines the effect of variation of the substitution pattern on the phenyl ring on the crystal structure of the compounds and the conformational changes that may take.

Experimental

Synthesis and Physical Measurements

Compounds **I–III** were synthesized by the addition of equimolar amounts of phthalic anhydride and the corresponding aniline derivatives under acetic acid reflux for 1 h with the progress of the reaction monitored by TLC (Scheme 1). Upon cooling, single crystals of the corresponding phthalimide derivatives suitable for X-ray diffraction were deposited. The crystals were filtered and washed several times with water. The synthesis is represented in scheme 1. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 300 MHz spectrometer using deuterated solvents and TMS as a reference operating at 300 and 75.5 MHz respectively. ^{1}H NMR (300 MHz): internal standard solvent DMSO-d_6 (2.49 ppm from TMS), internal standard TMS; ^{13}C NMR (75.5 MHz): internal standard solvent DMSO-d_6 (39.99 ppm from TMS): internal standard TMS; the splitting of proton resonances in the reported ^{1}H NMR spectra are defined as s = singlet, d = doublet and m = complex pattern. FT-IR spectra were recorded on Bio-Rad FTS 3000 MX spectrophotometer (400–4,000 cm^{-1}). The elemental analyses were conducted using a LECO-183 CHNS analyzer.

Crystal Structure Determination

X-ray data of the compounds were collected on a Rigaku RAXIS RAPID-II imaging plate diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.71075$ Å). The absorption corrections were carried out using a multi-scan method (ABSCOR) [12]. Cell refinement was carried out using PROCESS-AUTO and the data was reduced by using Crystal Structure [13]. Data collections with θ_{max} of 30^{o} were carried out at 190(2) K for all compounds. The structures were solved using SHELXS97 [14] and the structures were refined with SHELXL97 [14] by full-matrix least-squares on F^2 against all reflections. H atoms were treated as riding, with C–H = 0.95 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$. Molecular graphics were carried out using ORTEP-3 [15] and the material for publication prepared using MERCURY [16] and PLATON [17]. A summary of the crystal data and other details concerning data collection and structure refinement are given in Table1.

Results and Discussion

Structure Elucidation of Synthesized Compounds

*2-(3,4-Dichlorophenyl)isoindoline-1,3-dione (**I**)*

Yield 90 %; white solid; Elemental Analysis: Calc. for $C_{14}H_7Cl_2NO_2$: C, 57.56; H, 2.42; N, 4.79 Found: C, 57.53; H, 2.40; N, 4.83. FT-IR (KBr, cm^{-1}): 3080 Ph(CH), 1704(C=O), 1590(C–N), 1567(C–N), 1478, 1466, 1378, 1218, 1172, 1130, 1112, 1096, 1077, 1028, 888, 869, 831, 782 (C–Cl), 748, 711, 692, 679; ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.56 (d, 1H, $^{3}J_{HH}$ = 7.1 Hz, Ar*H*), 7.62 (d, 1H, $^{3}J_{HH}$ = 7.1 Hz, Ar*H*), 7.72 (s, 1H, Ar*H*), 7.90–8.12 (m, 4H, Ar*H*); ^{13}C NMR (75.5 MHz, DMSO-d_6) δ 123.2, 123.6, 127.9(2C), 129.6, 130.8, 131.9, 132.3(2C), 134.5, (Aromatic-*C*), 166.4(2C, C=O);

*2-(2,4-Dichlorophenyl)isoindoline-1,3-dione) (**II**)*

Yield 95 %; white solid; Elemental Analysis: Calc. for $C_{14}H_7Cl_2NO_2$: C, 57.56; H, 2.42; N, 4.79 Found: C, 57.51; H, 2.44; N, 4.82. FT-IR (KBr, cm^{-1}): 3084Ph(CH), 1715(C=O), 1585(C–N), 1568(C–N), 1485, 1467, 1373, 1221, 1170, 1145, 1112, 1095, 1084, 1055, 884, 872, 862, 788 (C–Cl), 773, 708, 685, 661; ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.66 (d, 1H, $^{3}J_{HH}$ = 7.1 Hz, Ar*H*), 7.68 (s, 1H, Ar*H*), 7.91 (d, 1H, $^{3}J_{HH}$ = 7.1 Hz, Ar*H*), 7.93-8.04 (m, 4H,

ArH); ^{13}C NMR (75.5 MHz, DMSO-d_6) δ 124.3(2C), 128.9, 129.2, 130.0, 131.8(2C), 133.1, 133.9, 135.5, 135.6(2C) (Aromatic-*C*), 166.6 (2C, C=O).

2-(2,4,5-Trichlorophenyl)isoindoline-1,3-dione) ***(III)***

Yield 90 %; white solid; Elemental Analysis: Calc. for $C_{14}H_6Cl_3NO_2$: C, 51.49; H, 1.85; N, 4.29 Found: C, 51.53; H, 1.86; N, 4.31. FT-IR (KBr, cm^{-1}): 3079 Ph(CH), 1710(C=O), 1588(C–N), 1572(C–N), 1481, 1468, 1375, 1216, 1168, 1132, 1110, 1092, 1074, 1026, 878, 864, 828, 780(C–Cl), 743, 716, 688, 676; ^{1}H NMR (300 MHz, DMSO-d_6) δ 7.56 (d, 1H, $^3J_{HH}$ = 6.9 Hz, ArH), 7.62 (d, 1H, $^3J_{HH}$ = 6.9 Hz, ArH), 7.72 (s, 1H, ArH), 7.90–8.12 (m, 4H, ArH); ^{13}C NMR (75.5 MHz, DMSO-d_6) δ 124.6, 127.3(2C), 130.2, 130.9, 131.9, 132.2(2C), 132.9(2C), 135.1, 135.5(Aromatic-*C*), 166.4(2C, C=O).

Crystal Structure Analysis

As summarized in Table 1, compounds **I** and **II** crystallize in a monoclinic crystal system with space group $P2_1/c$, while compound **III** crystallizes in a tetragonal crystal system with space group $I4_1/a$. Molecular structures of the compounds are presented in Fig. 1**a**–**c**. The three compounds differ with respect to the number and the positions of chlorine atoms on the phenyl ring. In all the three compounds, one chlorine atom occupies the *para* position. In compound **I**, there is an additional chlorine atom at the *meta* position, while in compound **II** the additional chlorine is at the *ortho* position. In compound **III**, however, there are two additional chlorine atoms occupying the *ortho* and *meta* positions. Some bond lengths and angles, and torsion angles are presented in Table 2. In all compounds, the nitrogen atoms adopt a near trigonal planar arrangement and no significant difference in all bond lengths and angles is observed except for the N1-C9 bond length in which a little difference is observed between **II** and *meta*-chloro-substituted compounds (**I** and **III**).

The phthalimide C1/N1/C2-C8/O1/O2 planes of all the compounds studied are nearly planar with *r.m.s.* deviations of 0.025(1), 0.024(1) and 0.025(2) Å, respectively, for compounds **I**, **II** and **III**. The maximum deviations from the mean planes are 0.0407(10) Å for O1 in compound **I**, 0.0435(11) Å for N1 in **II** and 0.029(2) Å for O2 in **III**. The phenyl rings of all the compounds twist out of the phthalimide plane with dihedral angles of 61.02(3), 69.09(3) and 85.78(5)°, respectively, for **I**, **II** and **III**, being comparable with those of 56.71(16)° [18] and 64.09(15)° [19] for *N*-phenylphthalimide. The progressive increase in the dihedral angle from compound **I**–**III** may suggest that the presence of the Cl at the *ortho* position leads to a greater degree of twist avoiding repulsion between the *ortho* Cl and carbonyl O atoms since **I** does not have a substituent in the *ortho* position. It would have been

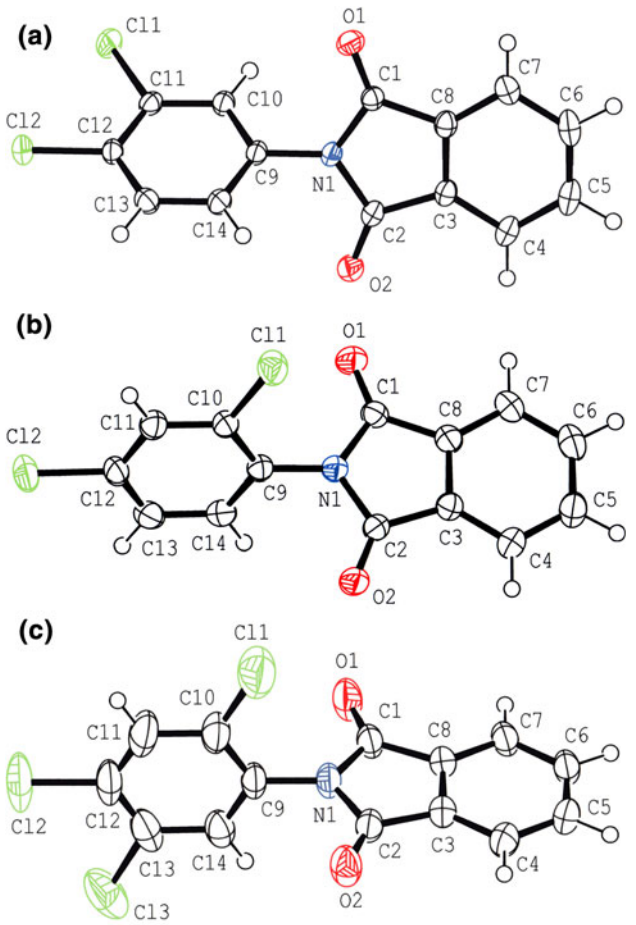


Fig. 1 Molecular structures of the compound **I** (**a**), **II** (**b**) and **III**(**c**), showing the atomic labelling scheme. Displacement ellipsoid for non-hydrogen atoms are plotted at the 50 % probability level

Table 2 Selected geometric parameters in compounds **I**, **II** and **III** (Å, °)

	I	II	III
N1–C1	1.4089(16)	1.4068(15)	1.403(2)
N1–C2	1.4148(15)	1.4141(15)	1.407(3)
N1–C9	1.4214(14)	1.4170(16)	1.423(2)
C1–O1	1.2092(15)	1.2057(15)	1.205(3)
C2-O2	1.2053(16)	1.2010(15)	1.203(3)
C1–N1–C9	124.34(10)	124.71(10)	124.03(17)
C2N1–C9	123.42(10)	123.07(10)	123.78(16)
N1–C9C10	119.38(11)	121.22(11)	121.0(2)
N1–C9–C14	119.11(10)	119.83(11)	119.6(2)
C1–N1–C9–C10	62.91(16)	−72.37(16)	−89.4(3)
C1–N1–C9–C14	−118.15(13)	108.88(14)	90.6(3)
C2–N1–C9–C10	−121.45(13)	112.77(13)	97.5(3)
C2–N1–C9–C14	57.49(16)	−65.98 (16)	−82.5(3)

Table 3 Hydrogen bond geometries (Å, °) for compounds **I**, **II** and **III**

	D–H	H···A	D···A	D–H···A
Compound **I**				
C4–H4···O2^{i}	0.95	2.53	3.3535(16)	145
C14–H14···O1^{ii}	0.95	2.34	3.2597(16)	162
Compound **II**				
C11–H11···O1^{i}	0.95	2.46	3.2229(17)	137
Compound **III**				
C4–H4···O1^{i}	0.95	2.37	3.195(3)	145
C7–H7···O2^{ii}	0.95	2.51	3.373(3)	151
C14–H14···O1^{iii}	0.95	2.48	3.247(3)	137

Symmetry codes: [**I**] (i) −x, −y, 1 − z, (ii) −1 + x, y, z; [**II**] (i) −x, 1 − y, 1 − z; [**III**] (i) x, −1/2 + y, 1 − z (ii) x, 1/2 + y, 1 − z; (iii) 1 − x, 3/2 − y, z

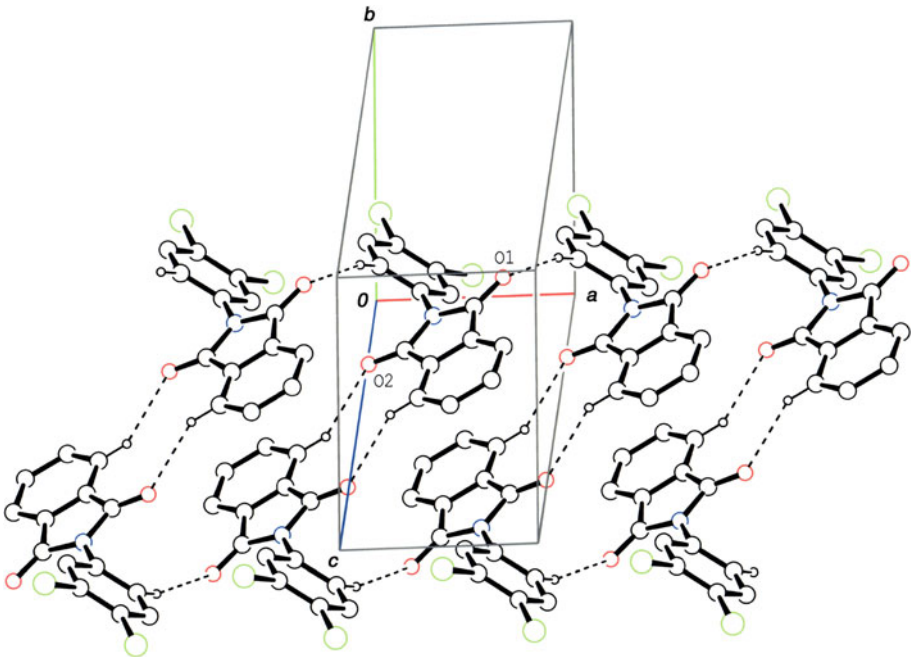


Fig. 2 Crystal packing diagram of compound **I**. The C–H···O hydrogen bonds are shown in *broken lines*

expected that the presence of bulky groups at the *ortho* position hence would increase the degree of twist if intramolecular repulsion are the predominant factors. Comparing the dihedral angles around this region between **II** and **III** however, the large difference observed is unexplainable only by the intramolecular repulsion alluded to earlier. In fact, the dihedral angles observed for *ortho*-substituted *N*-phenylphthalimide derivatives distribute in a wide range: 70.8(3), 88.24(10), 71.93(8), 59.99(5), 79.13(18), 84.74(13)°, 77.2(1)°, for methyl- [20], animo- [21], hydroxy- [22], methoxy- [23], bromo- [24], iode- [25] and ethyl-substituted [26] compounds respectively, which are poorly correlated with the size of the substituent since both bromine, iodine and ethyl group are far bigger than chlorine with the chlorine showing a bigger twist. It is obvious that both intra- and intermolecular interactions are responsible for the observed twist.

In the crystal structures of all compounds, the primary intermolecular interactions are C–H···O hydrogen bonds with the H···O contact distances and the C–H···O angles being in the ranges of 2.34-2.53 Å and 137–162°, respectively (Table 3). In compound **I**, the molecules are linked by a pair of the hydrogen bonds [C4–H4···O2^{i}; symmetry code (i) in Table 3] into a centrosymmetric dimer with an $R_2^2(10)$ graph-set motif [27]. The dimers are further connected by another hydrogen bond [C14–H14···O1^{ii}; symmetry code (ii) in Table 3], resulting in a double tape structure along the *a* axis (Fig. 2). The tapes are stacked along the *b* axis in the manner that the phthalimine planes are stacked anti-parallelly in close distances. The shortest

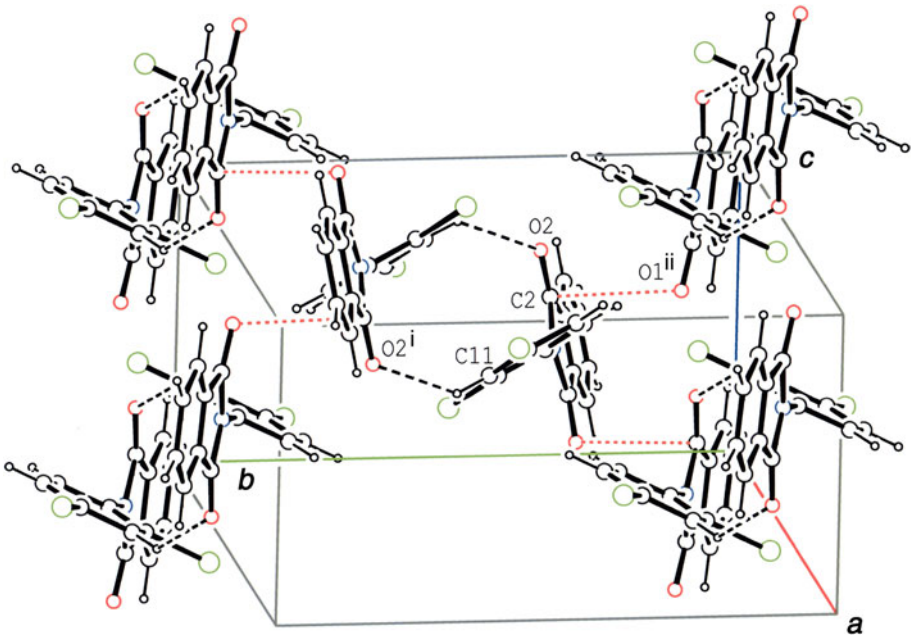


Fig. 3 Crystal packing diagram of compound **II**. The C–H···O hydrogen bonds and the C···O contacts are indicated by *black* and *red dashed lines*, respectively. Symmetry codes: (i) −x, 1 − y, 1 − z; (ii) x, 1/2 − y, 1/2 + z (Color figure online)

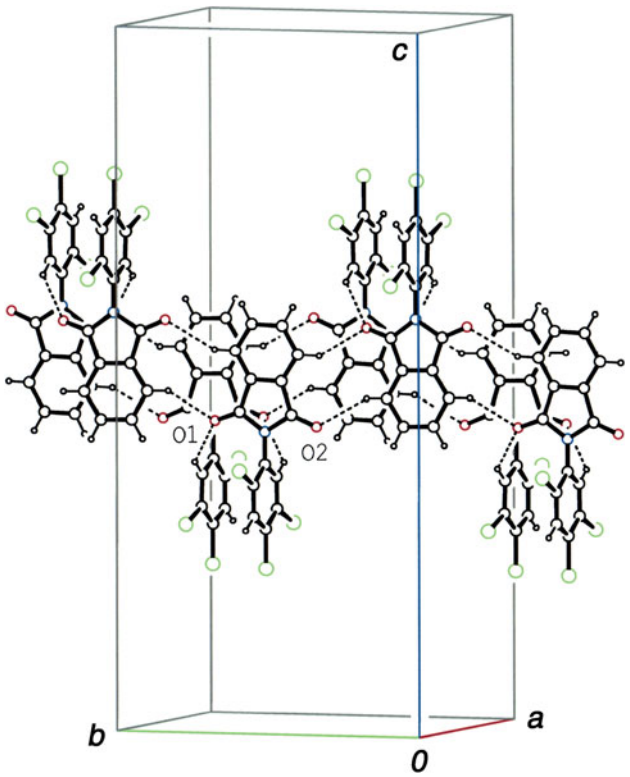


Fig. 4 Crystal packing diagram of compound **III**, showing a double-tape structure along the *b axis*. *Dashed lines* indicate C–H···O hydrogen bonds

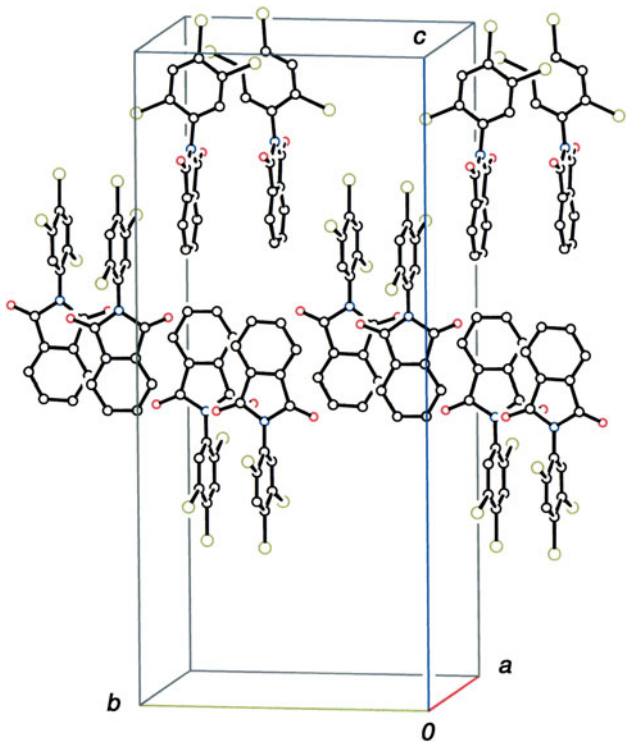


Fig. 5 Crystal packing diagram of compound **III**, showing the arrangement of double-tapes perpendicular to each other. H atoms have been omitted for clarity

C···C contact distances in the tape and between the tapes are C2···C5(1 − x, −y, 1 − z) = 3.3895(19) Å and C1···C7(1 − x, 1 − y, 1 − z) = 3.3263(18) Å.

Similar to **I**, the molecules in compound **II** are connected by a pair of C–H···O hydrogen bonds [C11–H11···O2^{i}: symmetry code (i) in Table 3] into a centrosymmetric dimer with an $R_2^2(14)$ graph-set motif. No further hydrogen bonding is observed but short C···O contacts [C2···O1(x, 1/2 − y, 1/2 + z) = 3.0717(16) and C3···O1(x, 1/2 − y, 1/2 + z) = 3.1512(16) Å] link the dimers into a layer parallel to the *bc* plane (Fig. 3).

In compound **III**, molecules are connected by C–H···O hydrogen bonds [C4–H4···O1^{i} and C7–H7···O2^{ii}; symmetry codes (i) and (ii) in Table 3], which form an $R_2^2(10)$ graph-set motif, into a tape running along the *b* axis. The tapes related by an inversion centre to each other are linked by another C–H···O hydrogen bond [C14–H14···O1^{iii}; symmetry code (iii) in Table 3], forming a double-tape structure (Fig. 4). Perpendicular to the tape, a symmetry equivalent neighboring double-tape related by a fourfold screw axis runs along the *a* axis (Fig. 5). Between the double-tapes, the phthalimide and chlorobenzene planes are arranged to be approximately parallel to each other and a Cl···Cl short contact [Cl1···Cl2(3/4 − y, 1/4 + x, 1/4 + z) = 3.3215(11) Å] is observed.

Conclusions

In this work three chloro-substituted derivatives of *N*-phenylphthalimide have been synthesized by refluxing equimolar amounts of phthalic anhydride and chlorine substituted anilines in acetic acid and characterized by elemental analysis, FT-IR and ^{1}H and ^{13}C NMR. The crystal structures were determined by single-crystal X-ray diffraction. The phthalimide moiety of all the compounds are coplanar even though some atoms deviate slightly from the mean plane when the least square parameters are analyzed. The phenyl ring in each case twists out of the phthalimide plane with angles of 61.02(3)^{o}, 69.09(3)^{o} and 85.78(5)^{o}, respectively, for compounds **I**, **II** and **III**. Few weak C–H···O interactions and short C···C, C···O and Cl···Cl contacts have been observed. In compound **I**, the molecules are linked by a pair of the hydrogen bonds into a centrosymmetric dimer with an $R_2^2(10)$ graph-set motif with these dimers further connected by another hydrogen bond into a double tape structure along the *a* axis. Compound **II** forms a centrosymmetric dimer with an $R_2^2(14)$ graph-set motif connected by a pair of C–H···O hydrogen bonds to each other. On the other hand, an $R_2^2(10)$ graph-set motif resulting from C–H···O hydrogen bonds and further connected by C–H···O hydrogen bonds into a tape running along the *b* axis are formed by compound **III**, molecules. The tapes related by an inversion centre to each other are linked by another C–H···O hydrogen bond into a double-tape structure.

Supplementary Data

Crystallographic data for the compounds **I**, **II** and **III** have been deposited with the Cambridge Crystallographic Centre, CDCC No. 885916, 885917 and 885918, respectively. The data can be obtained free of charge from CDCC, 12 Union Road, Cambridge CB2 1EZ, UK, Fax: +44 1223 336 033, e-mail: deposit@cdcc.cam.ac.uk or http: www.ccdc.cam.ac.uk.

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