A stereoscopic view of the crystal structure is shown in Fig. 4. The nitrophenyl groups stack along **b**. The angle between the nitrophenyl plane and **b** is 86·2 (2)° and that between the phthalimido plane and **c** is 59·6 (2)°. The mean distance between the nitrophenyl plane at (x, y, z) and that at (1−x, 2−y, 1−z) is 3·471 (5) Å. The packing contacts are of van der Waals type.

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SHORT COMMUNICATION

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1,4-Butanediyl bis(*p*-chlorobenzoate) (BDDP) and 1,6-hexanediyl bis(*p*-chlorobenzoate) (HDDP): further refinement in space group $P\bar{1}$.

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Abstract. The crystal structures of the title compounds, recently reported in space group $P1$, have been more satisfactorily refined in the centrosymmetric space group $P\bar{1}$ based on the data obtained from a new crystal for BDDP and the original data for HDDP. Whereas the original data for BDDP showed, for the $0kl$ reflections, significantly larger values of F_o than F_c, the new F_o values are smaller than before and in good agreement with the F_c's. The final parameters lead to more satisfactory geometries for both compounds.

Recently we described (Bocelli & Grenier-Loustalot, 1984*a*,*b*) the crystal structures of $C_{18}H_{16}Cl_2O_4$(BDDP) and $C_{20}H_{22}Cl_2O_4$(HDDP) in the noncentrosymmetric space group $P1$ [BDDP: $a = 7{\cdot}609$ (2), $b = 10{\cdot}318$ (1), $c = 5{\cdot}924$ (3) Å, $\alpha = 96{\cdot}31$ (2), $\beta = 98{\cdot}35$ (3), $\gamma = 111{\cdot}05$ (2)°, $Z = 1$; HDDP: $a = 10{\cdot}483$ (2), $b = 7{\cdot}760$ (2), $c = 6{\cdot}746$ (2) Å, $\alpha = 107{\cdot}77$ (2), $\beta = 98{\cdot}32$ (2), $\gamma = 70{\cdot}43$ (3)°, $Z = 1$]. Both structures were very nearly centrosymmetric, and we have now carried out more successful refinements in $P\bar{1}$.

BDDP: The earlier blocked-matrix refinement in $P1$ resulted in somewhat unusual geometry with, for example, the aromatic C—C distances ranging from 1·316 (9) to 1·494 (7) Å. Moreover, the observed intensities for the $0kl$ reflections were significantly larger than the calculated ones ($\sum |F_o| = 487{\cdot}5$, $\sum |F_c| = 322{\cdot}7$). Since the original crystal has gone, we are unable to check this phenomenon on Weissenberg photographs. Accordingly, we have collected intensity data from a new crystal.

The new crystal was prismatic, about 0·08 × 0·12 × 0·19 mm in size. Data were collected using the same experimental procedures as described earlier (Bocelli & Grenier-Loustalot, 1984*a*). Cell dimensions were: $a = 7{\cdot}612$ (2), $b = 10{\cdot}357$ (3), $c = 5{\cdot}926$ (2) Å, $\alpha = 96{\cdot}28$ (3),

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$\beta = 98{\cdot}05\ (2)$, $\gamma = 111{\cdot}32\ (3)°$. [The values of b, β and γ appear significantly different from before; both sets were obtained with the help of the program *CELFIT* (Calestani, 1985). It is probable that the differences are more realistic indicators of the experimental accuracy than the precision estimated in parentheses.] Of the 1556 independent reflections measured, 1239 were considered observed [$I > 2\sigma(I)$]. A detailed comparison of the new $F(0kl)$ values showed them to be systematically smaller than before, by a factor of about 0·6. Full-matrix refinement in the centrosymmetric space group $P\bar{1}$ quickly converged (max. $\Delta/\sigma = 0{\cdot}20$) at $R = 0{\cdot}057$ for 141 parameters; a similar R was obtained for the original data when the F_o values of the $0kl$ reflections were multiplied by 0·6. The final $P\bar{1}$ parameters are given in Table 1.† The conformation of the molecule is unchanged from the earlier $P1$ description but the bond lengths are now normal and the e.s.d.'s are smaller; the aromatic C—C distances range from 1·379 (4) to 1·396 (5) Å.

We do not know the cause for the peculiar $0kl$ measurements from the first crystal. The cell dimensions do not suggest that twinning, with the two $0kl$ nets coincident with the a^* axes in opposite directions, was a problem.

HDDP: Full-matrix refinement in space group $P\bar{1}$ converged (max. $\Delta/\sigma = 0{\cdot}21$) at $R = 0{\cdot}058$ for the original (Bocelli & Grenier-Loustalot, 1984*b*) 1455 reflections with $I > 2\sigma(I)$ and 158 parameters, compared with $R = 0{\cdot}060$ for 292 parameters in the earlier rigid-body refinement in $P1$. Atom coordinates are given in Table 2.† The range of aromatic C—C distances is 1·374 (4)–1·396 (3) Å; the conformation of the chain remains all-*trans*.

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates for both compounds have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42496 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and* B_{eq} *values (Hamilton, 1959) for BDDP*

	x	y	z	B_{eq}(Å^2)
Cl	9321 (1)	1453 (1)	7770 (1)	5·51
O(1)	3555 (3)	−4961 (2)	1851 (4)	5·03
O(2)	3091 (3)	−3577 (2)	−636 (3)	4·15
C(1)	5277 (4)	−2471 (3)	2885 (4)	3·40
C(2)	6098 (4)	−2557 (3)	5062 (5)	4·01
C(3)	7356 (4)	−1357 (3)	6582 (5)	4·26
C(4)	7777 (4)	−61 (3)	5848 (5)	3·91
C(5)	6994 (4)	49 (3)	3663 (5)	4·31
C(6)	5733 (4)	−1170 (3)	2192 (5)	3·98
C(7)	3911 (4)	−3811 (3)	1360 (4)	3·68
C(8)	1756 (5)	−4831 (3)	−2239 (6)	4·45
C(9)	711 (4)	−4366 (3)	−4093 (5)	4·00

Table 2. *Fractional atomic coordinates* ($\times 10^4$) *and* B_{eq} *values (Hamilton, 1959) for HDDP*

	x	y	z	B_{eq}(Å^2)
Cl	−4784 (1)	11255 (1)	3156 (1)	5·89
O(1)	1411 (2)	6574 (2)	6087 (2)	4·70
O(2)	1553 (2)	5267 (3)	2652 (3)	6·49
C(1)	−486 (2)	7641 (3)	3954 (3)	4·19
C(2)	−1189 (2)	8887 (3)	5700 (4)	4·58
C(3)	−2517 (2)	10026 (3)	5441 (4)	5·03
C(4)	−3111 (2)	9875 (3)	3467 (4)	4·48
C(5)	−2433 (3)	8659 (4)	1723 (4)	5·14
C(6)	−1108 (3)	7542 (3)	1972 (4)	5·07
C(7)	925 (2)	6361 (3)	4140 (3)	4·48
C(8)	2781 (2)	5368 (3)	6350 (3)	4·66
C(9)	3242 (2)	5913 (3)	8606 (3)	4·51
C(10)	4716 (2)	4709 (3)	8890 (3)	4·54

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