are planar and form angles of 12·9 (3) and 1·9 (2)° with the respective mean aromatic ring planes. The first value is larger than the angle of 7·5° measured for ethylene bis(*p*-chlorobenzoate) (Pérez & Brisse, 1975) which is the greatest reported in the literature for these compounds and is very near to that reported for the polyethylene terephthalate (12°) (Daubeny, Bunn & Brown, 1954).

Besides the torsion angles, the conformation of the C(9) and C(12) methyl groups may be described by the angles they form with the O(1), C(8), C(10) and O(3), C(11), C(10) and the O(2), C(7), O(1), C(8) and O(4), C(13), O(3), C(11) mean planes which are 32·4 (5), 143·0 (6), 50·8 (3) and 159·8 (4)°, respectively. Consequently, C(12) is *axial* and C(9) *equatorial* with respect to the chain.

The two carbonylic oxygens are *cis* with respect to the chain.

The crystal packing, which can be seen in Fig. 2, is determined by normal van der Waals interactions.

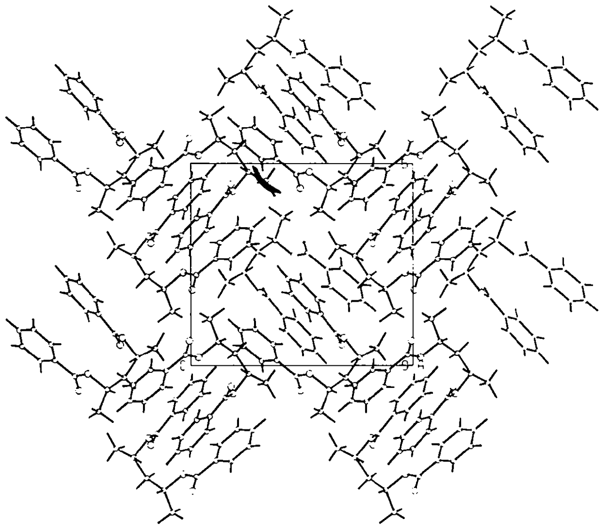

Fig. 2. Packing of the compound along the *x* axis.

The crystal structure determination of the title compound completes the *n*-methylene dibenzoate series ($n = 2,3,4$) for which the structures of oligomers, of di-methylated oligomers and of polymers are now available. Unfortunately, these data are not enough to offer a rationalization of the changes in the chain conformation which seems not univocally influenced by the presence of pendant methyl groups. This fact suggests that more conformational information is needed, and further crystallographic and spectroscopic work in this respect is in progress.

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Benzenehexacarbonitrile at 120 K, $C_{12}N_6$

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Abstract. $M_r = 228·176$, cubic, $Pa3$, $a = 10·781$ (6) Å, $V = 1253·1$ (1) Å^3, $Z = 4$, $D_x = 1·21$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $F(000) = 456$. Final $R = 0·048$ for the 'free-atom model' including 501 independent reflections. The molecule occupies one 4(*a*) position with $\bar{3}$ point symmetry of the space group. All

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six cyano groups point towards the center of a neighboring benzene ring, the distance between the N atom and the center being 3·657 (3) Å.

Introduction. The strong interactions between the aromatic system and the very electrophilic cyano group should show some effect in the charge-density distribution† of substituted aromatic compounds. Therefore 1,4-benzenedicarbonitrile (Drück, Guth, Kutoglu, Ohms, Scheringer & Heger, 1981) and benzenehexacarbonitrile have become the subjects of more sophisticated studies.

This paper reports the results of the conventional ('free-atom model') refinement of benzenehexacarbonitrile (henceforth abbreviated as HCB) using a data set collected at 120 (3) K.

Experimental. HCB crystallized in dry acetonitrile, slightly yellow octahedra, crystal 0·3 mm radius sealed in a thin glass capillary, mounted on a Philips PW1100 diffractometer, temperature slowly reduced.

The cooling device was an evaporator (Leybold Heraeus NCD-1) blowing cold nitrogen over the crystal. The nitrogen temperature, calibrated by the transition temperature of KH_2PO_4, was monitored throughout the experiment and showed no significant fluctuations.

Range of *hkl*: ±*hkl*; three standard reflections, intensity variation < 0·05 during whole measurement; 9737 reflections, $0{\cdot}073 \leq \sin\theta/\lambda \leq 0{\cdot}904$ Å^{-1}, Mo Kα, $\omega/2\theta$ scan mode; 1080 independent reflections, internal reliability factor for the 501 observed reflections $[I > 2\sigma(I)]$ being 0·050; no corrections for absorption and anomalous-dispersion effects; scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974).

The structure had been solved already by Littke (Littke & Wallenfels, 1965; Littke, 1967) for room-temperature film data but the results were unfortunately never published in detail.‡

Weighted least-squares refinement $[w = 1/\sigma^2(F)]$ starting with these parameters and an isotropic extinction factor gave $R(F) = 0{\cdot}048$, $R_w(F) = 0{\cdot}061$, GOF = 0·37 (29 parameters including 18 anisotropic temperature factors, 501 reflections), resulting extinction parameter $r^* = 8{\cdot}0 \times 10^{-4}$ (Finger & Prince, 1975); ratio of max. LS shift to error = 0·14; max. and min. heights in final difference Fourier map 0·34 and 0·27 e Å^{-3}; all computations performed on an IBM 370/145 computer at the Mineralogisches Institut der Universität Marburg, using special programs (Kutoglu, 1979) and parts of the XRAY system (Stewart, Kundell & Baldwin, 1970).

Discussion. Table 1 gives the positional and isotropic thermal parameters of the three independent atoms.*

Fig. 1 shows an *ORTEP* plot (Johnson, 1965) of the molecule and the resulting bond lengths and angles. The bond length in the aromatic ring is longer [Δd = 0·009 (2) Å] than the equivalent ones in 1,4-benzenedicarbonitrile [averaged bond length 1·388 (3) Å]. This lengthening is significant with respect to the e.s.d. The length of the bond to the cyano group corresponds well with the value reported for ethylenetetracarbonitrile [TCNE, averaged value 1·437 (3) Å; Drück & Guth, 1982] whereas the lengthening of the C—N triple bond [Δd = 0·005 (3) Å) compared with TCNE [averaged value 1·138 (3) Å] lies in the range of 2σ. The conventional refinements both in the structures of TCNE and HCB result in a physically meaningless shortening of the triple bond [HCB: 1·143 (3) Å]. When corrected for thermal motion (riding model), the resulting value of 1·161 (3) Å lies between the values of cubic [1·153 (2) Å; Little, Pautler & Coppens, 1971] and monoclinic [1·165 (3) Å] TCNE.

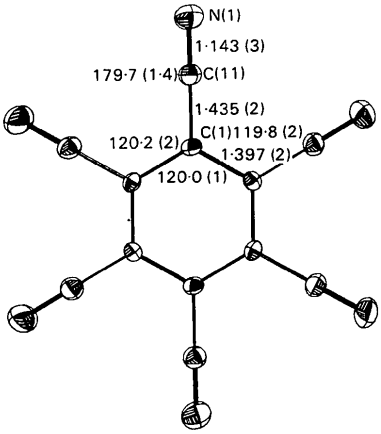

Fig. 1. *ORTEP* plot (50% probability) of HCB and bond lengths (Å) and angles (°) with the standard deviations (last digit) of the asymmetric unit.

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors*

$$B_{eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij}\, a^*_i\, a^*_j\, \mathbf{a}_i\, \mathbf{a}_j.$$

	x	*y*	*z*	B_{eq} (Å^2)
C(1)	937 (1)	−894 (1)	−46 (1)	1·13 (1)
C(11)	1902 (2)	−1809 (2)	−97 (2)	1·64 (1)
N(1)	2674 (2)	−2535 (2)	−142 (2)	2·75 (2)

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† Part of the work in the 'Sonderforschungsbereich 127. Kristallstruktur und Chemische Bindung' focuses on the determination of the electron density distribution in 'small' organic compounds.

‡ The space group was confirmed. The cell constants were redetermined by least-squares calculation *via* a selected number (16) of reflections.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38355 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

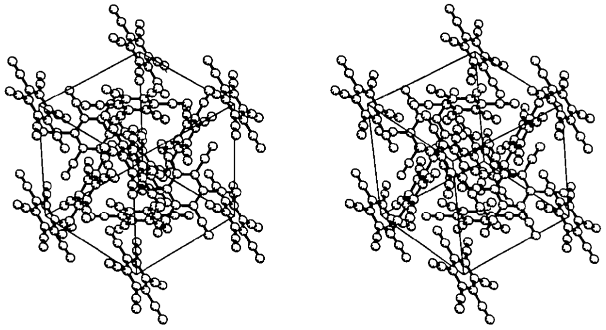

Fig. 2. Stereoscopic view [*PLUTO* 78 (Motherwell, 1978), modified by Drück, 1982] of the unit cell of HCB in the direction [1$\bar{1}$1].

Fig. 2 shows a stereoscopic view of the packing in the unit cell. The shortest intermolecular contacts are 3·148 (3) Å between a C atom of the ring and an N atom of the neighboring molecule and 3·230 (3) Å between the N and the C atom of a cyano group. With respect to the sum of the van der Waals radii (3·47 Å; Bondi, 1964) a considerable shortening of the mentioned intermolecular contacts is observed in the crystal structure. The cavity in the center of the unit cell, which can easily accommodate a small molecule, can be clearly observed.

The planarity of the whole molecule in the crystal structure is well defined by the molecular point symmetry $\bar{3}$ together with the calculated angles [90·1 (1), 90·1 (1) and 90·0 (1)°] between the body diagonal [111] and the lines from the molecule center to the atoms C(1), C(11) and N(1).* A remarkable fact is, that each cyano group is pointing almost linearly towards the center of a neighboring molecule, the corresponding distance [N(1)—center] and angle [C(11)—N(1)—center] being 3·657 (3) Å and 174·2 (5)° respectively. Considering the high melting point of HCB (643 K accompanied by decomposition), we can postulate an intermolecular charge compensation from the electronegative cyano group over its lone pair into the electron-deficient aromatic ring of a neighboring molecule. It could even be possible that, similar to reported results for biphenyl and hexachlorobenzene (Kitaigorodskii, 1965), the ring of benzenehexacarbonitrile is planar only in the crystal, whereas in the gas phase the strong electron-pulling forces of the six cyano groups cause a more 'cyclohexane-like' conformation of the benzene ring.

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* The positional deviations for the mentioned atoms from the best least-squares plane calculated including the six ring atoms are 0·002 Å.

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3,4-Dihydro-2-diphenymethyl-4-diphenylmethylene-6-methyl-3-(*p*-tolyl)quinazoline, $C_{42}H_{34}N_2$

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Abstract. M_r = 566·74, triclinic, $P\bar{1}$, a = 12·998 (6), b = 13·191 (4), c = 21·864 (3) Å, α = 97·53 (3), β = 90·74 (3), γ = 118·44 (5)°, V = 3255·8 Å^3, Z = 4, D_x = 1·15 Mg m^{-3}, Mo $K\alpha$, λ = 0·71069 Å, μ = 0·072 mm^{-1}, T = 293 K, R = 0·093 for 3668 reflexions [$F > 3\,\sigma(F)$]. The asymmetric unit consists of two molecules of the title compound related by an approximate non-crystallographic twofold axis. Bulky substituents prevent the quinazoline skeleton achieving planarity.

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