Fig. 1. Stereoscopic drawing of the unit cell of $Cs_2[Pt(CN)_4].H_2O$.

The average Pt—C bond length is 1·988 (7) Å, and the average C—N bond length is 1·160 (8) Å. The Pt—C—N angle is nearly linear with a 178·76 (60)° average. The four C atoms surrounding the Pt atom and the attached N atoms are planar with a maximum deviation of 0·006 Å, while the Pt atom is exactly in this plane. The dihedral angle between adjacent $Pt(CN)_4^{2-}$ groups is 13·0 (2)°.

The coordination sphere around each Cs atom is listed in Table 2. The coordination is sevenfold around Cs(2) and eightfold around Cs(1). All Cs—N distances are longer than the van der Waals sum (~3·1 Å). Apparently, the $Cs^+ \cdots ^-N \equiv C$ interactions are not of sufficient strength to distort the $Pt(CN)_4^{2-}$ groups. The O of the water molecule is 3·155 (8) Å from Cs(1) and 3·125 (9) Å from Cs(2).

The water molecule has an intramolecular bond angle [H(1)—O(1)—H(2)] of 104·4 (9)°. The O(1)—H(1) distance is 0·953 (10) Å and O(1)—H(2) is 0·957 (12) Å. If a riding-motion correction is made, O—H distances become 0·965 (11) and 0·985 (13) Å respectively.

Both H(1) and H(2) form hydrogen bonds to N(4) and N(2) respectively. The distances are 1·866 (9) and 2·168 (10) Å for H(1)—N(4) and H(2)—N(2). O(1)—H(1)—N(4) is 174·5 (10)° and O(1)—H(2)—N(2) is 168·0 (12)°.

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N,N'-Bis(β-chloroethyl)pimelamide*

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Abstract. $C_{11}H_{20}N_2O_2Cl_2$, M_r = 283·15, monoclinic, $P2_1$, a = 4·941 (5), b = 32·425 (30), c = 4·817 (5) Å, β = 113°37 (5)' at 25 °C, V = 707·07 Å³, D_x = 1·331 g cm⁻³, Z = 2. The molecular conformation is not fully extended. Each molecule forms hydrogen bonds along two directions (almost the a and c directions). A com-

* Model Compounds of Nylons. III.

parison with *N,N'*-bis(β-chloroethyl)glutaramide is made.

Introduction. The title compound (NNCEP) was prepared as described in the literature (Gladych & Taylor, 1962). Weissenberg photographs showed systematic absences consistent with the monoclinic space group $P2_1$ (0*k*0 with *k* odd). A colourless platelet was used to collect intensity data (Ni-filtered Cu *Kα* radiation, λ = 1·5418 Å) on a Siemens automatic single-crystal diffractometer. 1279 independent reflexions were collected and corrected for polarization and Lorentz factors. 122 of these reflexions were not considered in the refinement since their values were less than $3\sigma(I)$. The structure was solved by the Patterson method. The heaviest interatomic vectors on the Harker section were interpreted and, accordingly, the positions of the two independent Cl atoms were derived. The Fourier map, calculated by phasing the reflexions with only these two atoms, revealed all the remaining atoms except H. Refinement was achieved by least-squares procedures with anisotropic thermal factors for all except the H atoms. Their positions were calculated stereochemically and isotropic thermal factors equal to those of the carrier atoms were assigned to each of them. These parameters were kept fixed in the refinement. The weighting scheme suggested by Cruickshank & Pilling (1961) was applied; atomic scattering factors were calculated according to Moore (1963). The refinement was ended when the maximum shifts in the atomic coordinates and anisotropic thermal factors were less than $\frac{1}{5}$ and $\frac{1}{3}$ of the corresponding standard deviations respectively. The final *R* value is 0·069.

Table 1 contains the final positional parameters of NNCEP, with their estimated standard deviations.*

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32394 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. Solid-state studies of conformations and crystal structures of model compounds for synthetic polyamides (nylons) have been previously reported from our laboratories (Benedetti, Ciajolo & Corradini, 1973, 1974).

Data on two compounds of general formula:

$$R-(CH_2)_2-NH-CO-(CH_2)_n-CO-NH-(CH_2)_2-R$$

have given unexpected results as far as the conformation of the CH_2 chain between the two amide groups is concerned. The two compounds studied, *N,N'*-diethyladipamide (*n* = 4, *R* = H) and *N,N'*-bis(β-chloroethyl)glutaramide (NNCEG) (*n* = 3, *R* = Cl), show a succession of internal rotation angles for the bonds between the two amide groups of the type: skew(+), gauche(−), trans, gauche(+), skew(−) and trans, trans, gauche(+), trans, respectively. These conformations were probably the result of the different system of hydrogen bonds present in the two series of
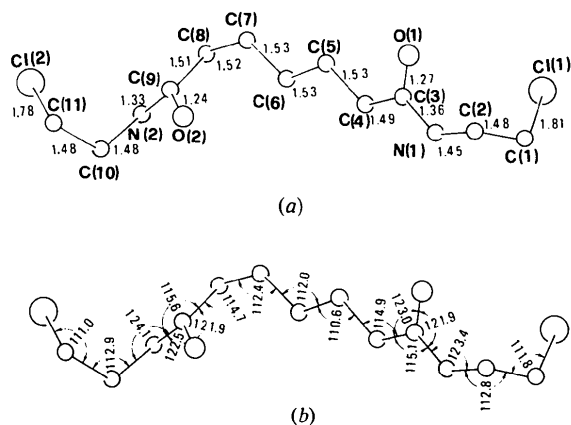

Fig. 1. Molecular model of NNCEP with (*a*) bond lengths (Å) and (*b*) bond angles (°). The mean standard deviations of bond lengths and bond angles are less than 0·03 Å and 0·5° respectively.

Table 1. *Final positional parameters* ($\times 10^4$)

	x	*y*	*z*
Cl(1)	8137 (4)	5000 (0)	−1817 (5)
Cl(2)	16478 (4)	801 (1)	−230 (5)
O(1)	6845 (12)	3916 (2)	688 (9)
O(2)	10685 (8)	1895 (2)	−1441 (11)
N(1)	5574 (11)	4111 (2)	−4249 (10)
N(2)	14666 (10)	1712 (1)	−2322 (11)
C(1)	4659 (16)	4851 (2)	−4791 (16)
C(2)	3651 (12)	4445 (2)	−4197 (14)
C(3)	7071 (11)	3869 (2)	−1818 (12)
C(4)	8961 (14)	3546 (2)	−2333 (13)
C(5)	10351 (11)	3243 (2)	279 (13)
C(6)	11958 (13)	2898 (2)	−616 (12)
C(7)	13527 (12)	2601 (2)	2015 (12)
C(8)	15231 (11)	2266 (2)	1183 (13)
C(9)	13321 (10)	1944 (2)	−989 (12)
C(10)	13261 (15)	1361 (2)	−4343 (14)
C(11)	12958 (14)	992 (2)	−2685 (19)

Table 2. *Internal rotation angles* (°)

trans conformation = 180°.

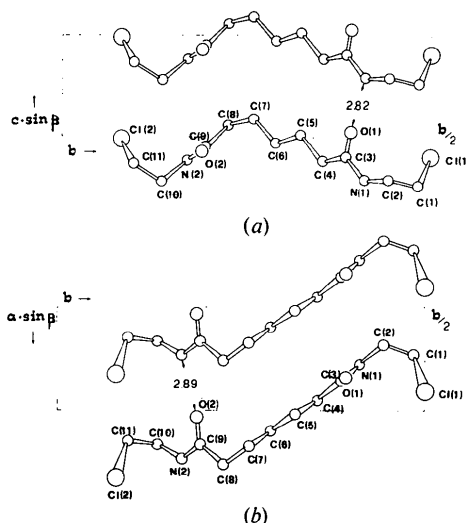|	
Cl(1)—C(1)—C(2)—N(1)	−59
C(1)—C(2)—N(1)—C(3)	103
C(2)—N(1)—C(3)—O(1)	1
C(2)—N(1)—C(3)—C(4)	181
N(1)—C(3)—C(4)—C(5)	187
C(3)—C(4)—C(5)—C(6)	173
C(4)—C(5)—C(6)—C(7)	178
C(5)—C(6)—C(7)—C(8)	182
C(6)—C(7)—C(8)—C(9)	−55
C(7)—C(8)—C(9)—O(2)	−19
C(7)—C(8)—C(9)—N(2)	162
C(8)—C(9)—N(2)—C(10)	176
C(9)—N(2)—C(10)—C(11)	269
N(2)—C(10)—C(11)—Cl(2)	−47
C(5)—C(4)—C(3)—O(1)	6
C(10)—N(2)—C(9)—O(2)	−3

Fig. 2. Projections of NNCEP along (a) a and (b) c. Hydrogen bonds are indicated.

compounds, having respectively an even or an odd number of methylene groups between the carbonyls of the amide groups.

In order to gain more information on this class of compounds and confirm the previous results, we have undertaken the X-ray analysis of the crystal structure of NNCEP ($n = 5, R = Cl$).

The molecular model of NNCEP is given in Fig. 1 with bond lengths and bond angles; in Table 2 are reported the internal rotation angles of NNCEP.

All the geometric parameters of the molecule are consistent with known literature data on amide groups and paraffin chains. The two amide groups are nearly planar: the root-mean-square distance of the atoms from the best plane passing through them is less than 0·03 Å for both the independent amide groups. The molecular conformation is not fully extended. As in the case of NNCEG, the other derivative with an odd number of methylene groups between the carbonyls of the amide groups, one *gauche* C—C bond is present in the dicarboxylic acid moiety. Again, this conformation is

one of the not fully extended conformations of minimum internal energy which allows for the formation of two hydrogen bonds along two different directions (almost the a and c directions) giving rise to bidimensional layers (parallel to the ac plane with width b/2) of hydrogen-bonded molecules [the lengths of the two hydrogen bonds $N(1)\cdots O(1)$ and $N(2)\cdots O(2)$ are 2·82 and 2·89 Å].

This result was to be expected also on the basis of the unit-cell constants of NNCEP as compared with those of NNCEG: in fact the lengths of the a and c axes for the two compounds are conservative, as well as the β angles, while the b axis increases from 28·123 Å (for NNCEG) to 32·425 Å (for NNCEP), the increase of 4–5 Å being due to two additional CH$_2$'s per molecule.

The conformation of the molecule at its termini described by the internal rotation angles Cl(1)—C(1)—C(2)—N(1), Cl(2)—C(11)—C(10)—N(2), C(1)—C(2)—N(1)—C(3) and C(11)—C(10)—N(2)—C(9) is very close to that observed for NNCEG and as previously pointed out (Benedetti, Ciajolo & Corradini, 1974), must be favoured by dipole–dipole interactions between different molecules belonging to adjacent layers of hydrogen-bonded molecules.

The modes of packing for NNCEP on two projections along the a and c directions are shown in Fig. 2.

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