The structure was discovered with *MULTAN*80 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Initial atomic positions were refined by full-matrix least squares minimizing the function $\sum w(|F_o| - |F_c|)^2$. The weights, w, were defined as $4F_o^2/S^2(I)$ with $S^2(I) = [\sigma^2(I_c) + (0{\cdot}06\,F_o)^2]$. Non-H atoms were refined with anisotropic thermal parameters. Positions for all of the H atoms were determined from successive difference Fourier maps and, except for those attached to the Boc group which were held at fixed calculated positions with fixed isotropic temperature factors, were refined along with the isotropic temperature factors. The refinement converged; max. Δ/σ in the final cycle was 0·03. The standard crystallographic agreement factors were $R = 0{\cdot}039$ and $wR = 0{\cdot}051$, GOF = 1·23 for 1757 observations and 281 variables. An extinction coefficient of the kind described by Zachariasen (1963) refined in the latter stages to $3{\cdot}89\,(1) \times 10^{-7}$. A final difference Fourier map showed maximum positive and negative excursions of 0·153 and 0·129 e Å^{-3}. A refinement using all 2419 data with $I \geq 0{\cdot}01\sigma(I)$ gave $R = 0{\cdot}056$, $wR = 0{\cdot}054$. Values of the neutral-atom scattering factors were employed as implemented in the Enraf–Nonius *SDP* (Frenz, 1987), a locally modified version of which was the source of all programs. Atomic coordinates are presented in Table 1.* Bond distances and angles are listed in Table 2. A view of the molecule is provided as Fig. 1.

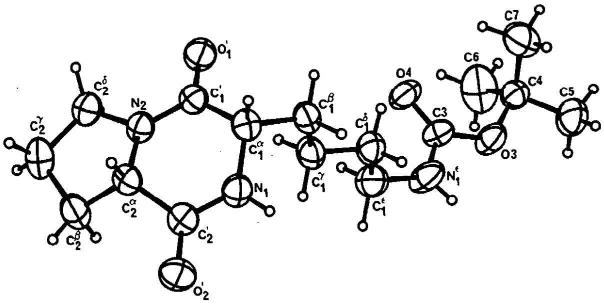

Fig. 1. *ORTEP* (Johnson, 1976) drawing of *cyclo*(-*N*-ε-Boc-L-Lys-L-Pro-). Non-H atoms are shown as principal ellipses at the 50% probability level, H atoms as small spheres of arbitrary size.

Related literature. Structures and conformations of diketopiperazines have been the subject of multitudinous publications. Much of this work has been reviewed and analyzed in a series of papers (Jankowska & Ciarkowski, 1987; Gdaniec, Liberek, Kolodziejczyk, Jankowska & Ciarkowski, 1987; Karle, 1979). Proline ring conformations have been analyzed and categorized by Ashida & Kakudo (1974).

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* Lists of structure factors, H-atom positions, bond distances and angles involving hydrogen and anisotropic librational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52675 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of Two Nitro-Substituted Aryl Carbonates

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Abstract. 2-Nitrophenyl 2-nitrophenoxyformate (I), $C_{13}H_8N_2O_7$, $M_r = 304{\cdot}2$, monoclinic, Pc, $a = 7{\cdot}887\,(3)$, $b = 10{\cdot}969\,(7)$, $c = 8{\cdot}113\,(5)$ Å, $\beta = 115{\cdot}11\,(4)°$, $V = 635{\cdot}5$ Å^3, $Z = 2$, $D_x = 1{\cdot}590$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}2$ cm^{-1}, $F(000) = 312$, $T = 173$ K, $R = 0{\cdot}0396$ for 1497 unique reflections

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Table 1. *Experimental details*

	(I)	(II)
Crystal		
habit	Plate	Needles
size (mm)	0·15 × 0·15 × 0·05	0·60 × 0·05 × 0·05
Lattice parameters		
No. reflections	20	23
2θ range (°)	15·9–41·9	6·9–29·9
Reflection range		
h	−6 to 10	−13 to 13
k	−2 to 14	−22 to 0
l	−10 to 10	0 to 10
Max. $\sin\theta/\lambda$ (Å^{-1})	0·650	0·650
Check reflections	$0\bar{2}\bar{1}$ 230 $3\bar{2}\bar{1}$	$0\bar{4}1$ 031 $0\bar{8}0$
variation (%)	2 3 4	3 4 4
Reflections		
collected	1650	3735
unique observed	1497	2554
R_{int}	0·0000	0·0249
Observed criterion	$I > 2\sigma(I)$	$I > 2\sigma(I)$
No. parameters	198	254
R	0·0396	0·0671
wR	0·0593	0·0579
S	0·68	1·46
Secondary-extinction parameter (χ)	0·010 (2)	0·0017 (3)
$F^* = F[1 + 0{\cdot}002\chi F^2/\sin(2\theta)]^{-1/4}$		
Weighting factor (*g*)	0·00071	0·0003
$w^{-1} = \sigma^2(F) + gF^2$		
Fourier difference peaks		
min. (e Å^{-3})	−0·26	−0·37
max. (e Å^{-3})	0·30	0·46
Max. Δ/σ	0·001	0·001

with $I > 2\sigma(I)$. 2,4-Dinitrophenyl 2,4-dinitrophenoxyformate (II), $C_{13}H_6N_4O_{11}$, $M_r = 394{\cdot}2$, monoclinic, $P2_1/c$, $a = 10{\cdot}682$ (4), $b = 16{\cdot}968$ (6), $c = 8{\cdot}243$ (3) Å, $\beta = 99{\cdot}57$ (9)°, $V = 1473{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}775$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}5$ cm^{-1}, $F(000) = 800$, $T = 185$ K, $R = 0{\cdot}0671$ for 2554 unique reflections with $I > 2\sigma(I)$. (I) and (II) are (nearly) symmetric molecules of nitro-substituted aryl carbonates. An O atom on each of the *ortho*-nitro groups is oriented both towards and proximal to the carbonyl carbon; these non-bonding O–C distances average 2·754 (20) Å (I) and 2·652 (55) Å (II).

Table 2. *Atom coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3) *for* (I)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
N(1)	−534 (5)	3217 (2)	−384 (5)	239 (8)
N(2)	161 (5)	9060 (2)	−682 (5)	247 (8)
O(1)	−81 (5)	6437 (2)	−1482 (5)	257 (7)
O(2)	1088 (5)	5498 (2)	1282 (4)	225 (6)
O(3)	−878 (5)	2132 (2)	−297 (5)	399 (10)
O(4)	−1715 (5)	4022 (2)	−841 (5)	294 (7)
O(5)	−974 (5)	6874 (2)	779 (4)	224 (6)
O(6)	377 (5)	9729 (2)	−1786 (5)	386 (10)
O(7)	1445 (5)	8596 (2)	636 (5)	312 (8)
C(1)	0	6288 (2)	0	189 (8)
C(2)	2133 (5)	4674 (2)	776 (5)	196 (8)
C(3)	3974 (5)	4943 (3)	1202 (5)	245 (9)
C(4)	5078 (5)	4106 (3)	819 (5)	289 (10)
C(5)	4346 (6)	2991 (3)	46 (5)	280 (9)
C(6)	2522 (6)	2684 (3)	−308 (5)	248 (9)
C(7)	1418 (5)	3539 (2)	50 (5)	203 (8)
C(8)	−2210 (5)	7786 (2)	−185 (5)	200 (8)
C(9)	−4022 (6)	7666 (3)	−331 (5)	262 (10)
C(10)	−5351 (5)	8553 (3)	−1196 (6)	338 (11)
C(11)	−4895 (6)	9554 (3)	−1954 (5)	325 (10)
C(12)	−3095 (6)	9693 (3)	−1841 (5)	290 (10)
C(13)	−1749 (5)	8815 (2)	−918 (5)	205 (8)

Table 3. *Atom coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3) *for* (II)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
N(1)	3143 (2)	5927 (1)	−2473 (3)	194 (7)
N(2)	4559 (2)	3516 (1)	−4810 (3)	255 (8)
N(3)	440 (2)	5083 (1)	3241 (3)	207 (7)
N(4)	−1318 (2)	7658 (2)	4229 (3)	279 (9)
O(1)	1353 (2)	5362 (1)	−59 (3)	279 (7)
O(2)	3440 (2)	5081 (1)	739 (2)	193 (6)
O(3)	3229 (2)	6345 (1)	−1253 (3)	277 (7)
O(4)	2814 (2)	6163 (1)	−3880 (3)	265 (7)
O(5)	4292 (2)	3824 (1)	−6171 (3)	322 (8)
O(6)	5124 (2)	2891 (1)	−4543 (3)	340 (8)
O(7)	2574 (2)	5925 (1)	2146 (3)	222 (6)
O(8)	1416 (2)	4696 (1)	3286 (3)	309 (8)
O(9)	−593 (2)	4800 (1)	3376 (2)	246 (7)
O(10)	−2162 (2)	7305 (1)	4763 (3)	439 (9)
O(11)	−1207 (2)	8376 (1)	4264 (3)	456 (10)
C(1)	2328 (3)	5446 (2)	829 (3)	192 (9)
C(2)	3573 (2)	4707 (2)	−728 (3)	174 (8)
C(3)	3484 (3)	5090 (2)	−2242 (3)	174 (8)
C(4)	3766 (2)	4695 (2)	−3598 (3)	179 (8)
C(5)	4176 (3)	3923 (2)	−3398 (4)	195 (9)
C(6)	4259 (3)	3531 (2)	−1922 (4)	211 (9)
C(7)	3932 (3)	3924 (2)	−587 (4)	212 (9)
C(8)	1532 (3)	6322 (2)	2560 (3)	192 (8)
C(9)	516 (3)	5946 (2)	3096 (3)	188 (8)
C(10)	−444 (3)	6379 (2)	3612 (4)	204 (9)
C(11)	−346 (3)	7192 (2)	3587 (4)	226 (9)
C(12)	643 (3)	7582 (2)	3047 (4)	239 (9)
C(13)	1588 (3)	7136 (2)	2515 (4)	232 (9)

Experimental. Compound (I) obtained by reaction of phosgene with 2-nitrophenol and two equivalents of pyridine, recrystallized from CH_2Cl_2/hexane solution at 253 K. Compound (II) obtained by reaction of phosgene with 2,4-dinitrophenol and two equivalents of pyridine, recrystallized from CH_2Cl_2/hexane solution at 253 K; poorly diffracting (hence higher *R* value). Compounds (I) and (II) were sealed in glass capillaries under dry nitrogen due to their moisture sensitivity. Siemens *R*3*m*/*V* upgrade of Nicolet *P*3*F* automated diffractometer, 2θ–θ scan with variable scan speeds. D_m not determined. Both structures were solved by direct methods using the *SHELXTL-Plus* (MicroVAX II) program package (Sheldrick, 1988). H atoms located by difference map and constrained to have C—H = 0·96 Å and isotropic thermal parameters, $U = 0{\cdot}08$ Å^2. All non-H atoms treated as anisotropic. No absorption correction necessary for either structure. Details of the data collection for both structures are in Table 1. Scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic coordinates for (I)

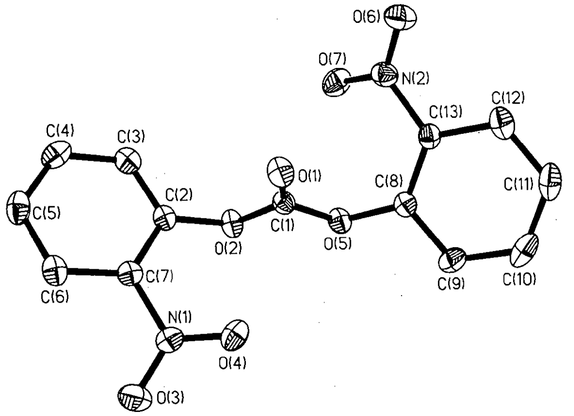

Fig. 1. Thermal-ellipsoid (50% probability) plot of (I).

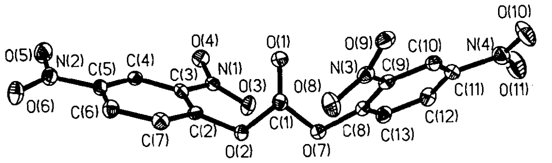

Fig. 2. Thermal-ellipsoid (50% probability) plot of (II).

and (II) are contained in Tables 2 and 3.[*] Figs. 1 and 2 illustrate the molecules with the numbering schemes employed.

Related literature. For additional information on related carbonate structures and chemistry, see Bendler (1985), Klug (1955), Perez & Scaringe (1987) and Henrichs & Luss (1988).

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[*] Lists of structure factors, anisotropic thermal parameters, bond lengths and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52704 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of α-Truxillamide Monohydrate

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Abstract. 2,4-Diphenyl-1,3-cyclobutanedicarboxamide monohydrate, $C_{18}H_{18}N_2O_2.H_2O$, $M_r = 312{\cdot}18$, m.p. 551–553 K, orthorhombic, *Pbcn*, $a = 16{\cdot}710\,(4)$, $b = 21{\cdot}111\,(2)$, $c = 8{\cdot}901\,(1)$ Å, $V = 3139{\cdot}9\,(8)$ Å^3, $Z = 8$, $D_m = 1{\cdot}32\,(2)$, $D_x = 1{\cdot}321$ Mg m^{-3}, Cu $K\alpha(\lambda = 1{\cdot}54178$ Å), $\mu = 0{\cdot}75$ mm^{-1}, $F(000) = 1328$, $T = 295$ K, $R = 0{\cdot}069$ for 1988 unique reflections with $|F_o|$ larger than $2\sigma(F_o)$. Cinnamamide has been ascertained to be photodimerizable in the crystal form grown from an ethanol solution and the crystal structure of a monohydrate of the photodimer has been determined.

Experimental. Crystals of the monomer, cinnamamide, were grown from an ethanol solution by slow evaporation. The Weissenberg photographs of the crystals showed that the space group, lattice constants and intensity distribution are the same as those of the crystals grown from a chloroform solution (Iwamoto, Kashino & Haisa, 1989*a*). Photodimerization was effected by exposing the monomer crystals to sunlight for two months (the method used by Cohen, Schmidt & Sonntag, 1964). At first, the crystals of the anhydrate of the photodimer ($P2_1/a$, $Z = 4$) (Vaida, Shimon, van Mil, Ernst-Cabrera, Addadi, Leiserowitz & Lahav, 1989) were grown from an ethanol solution of the irradiated sample by slow evaporation. The crystals of the monohydrate of the photodimer were obtained from an ethanol solution of the anhydrate by recrystallization. D_m by flotation in aqueous KI. A colorless prism elongated along **c**; $0{\cdot}12 \times 0{\cdot}07 \times 0{\cdot}30$ mm. Systematic absences $0kl$ k odd, $h0l$ l odd, $hk0$ $h + k$ odd, space group

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