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1,2,3-Triphenyl-5-(1,2-diphenyl-2-phenyliminoethylidene)-2-pyrrolin-4-one

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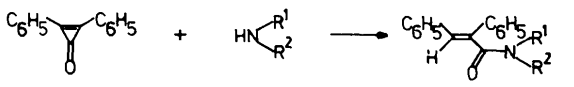
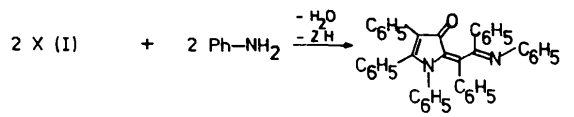
Abstract. $C_{42}H_{30}N_2O$, $M_r = 578{\cdot}7$, triclinic, $a = 10{\cdot}164\ (3)$, $b = 10{\cdot}290\ (3)$, $c = 16{\cdot}550\ (3)$ Å, $\alpha = 107{\cdot}13\ (5)$, $\beta = 96{\cdot}38\ (5)$, $\gamma = 104{\cdot}82\ (5)°$, $U = 1566{\cdot}2$ Å^3; $Z = 2$, $D_c = 1{\cdot}227$ Mg m^{-3}; $F(000) = 608$, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}54178$ Å, μ(Cu $K\alpha$) = 0·493 mm^{-1}. Space group $P\bar{1}$. Final $R = 0{\cdot}061$ for 2458 unique X-ray diffractometer data. The molecular structure has been elucidated.

Introduction. The reaction of diphenylcyclopropenone (I) with primary and secondary amines has been shown to yield (*E*)-α,β-diphenylacrylic amides (II) in a clear-cut nucleophilic opening of the three-membered ring (Eicher & Weber, 1975). Aniline, however, gave the expected acrylic amide (II, $R^1 = H$, $R^2 = Ph$) only as a minor product. Instead, a red compound, m.p. 473–474 K (from glycol monomethyl ether), was isolated as the main product (Chatila, 1976), whose composition (see above) revealed that two molecules of (I) and two of aniline had reacted in a complex dehydration–dehydrogenation process, the mechanistical evaluation of which is currently under investigation. Since degradation by hydrolysis, oxidation or reduction proved to be unsuccessful, the structure elucidation was achieved by X-ray analysis, which showed the unusual reaction product of (I) and aniline to be the title compound (III).



(I) (II)



(III)

The intensities of 2458 unique reflexions ($I > 3\sigma$) with $3 < \theta < 57°$ were measured by the $\omega/2\theta$ scan

Table 1. *Positional parameters* (×10⁴) *with e.s.d.'s in parentheses*

	x	*y*	*z*
N(1)	3932 (6)	7135 (6)	3436 (4)
C(2)	4551 (8)	7417 (7)	4295 (5)
C(3)	5968 (8)	7823 (7)	4430 (5)
C(4)	6344 (9)	7723 (8)	3591 (5)
O(41)	7497 (8)	7987 (7)	3406 (5)
C(5)	4992 (8)	7267 (7)	2958 (5)
C(6)	4880 (7)	7133 (7)	2109 (5)
C(7)	6097 (8)	6974 (9)	1683 (4)
N(8)	6387 (6)	5804 (7)	1425 (5)
C(11)	2515 (8)	6313 (8)	3067 (5)
C(12)	2224 (9)	4944 (9)	2472 (5)
C(13)	845 (9)	4167 (9)	2098 (6)
C(14)	−213 (10)	4734 (10)	2318 (7)
C(15)	83 (9)	6096 (9)	2915 (6)
C(16)	1467 (9)	6894 (9)	3297 (6)
C(21)	3629 (8)	7271 (8)	4915 (5)
C(22)	2819 (8)	5925 (9)	4876 (5)
C(23)	1897 (9)	5822 (9)	5438 (6)
C(24)	1761 (10)	7036 (10)	6022 (6)
C(25)	2588 (9)	8360 (9)	6065 (6)
C(26)	3523 (9)	8480 (9)	5524 (6)
C(31)	6992 (8)	8248 (8)	5229 (5)
C(32)	8264 (9)	9283 (9)	5361 (6)
C(33)	9279 (9)	9678 (9)	6108 (6)
C(34)	9033 (10)	9040 (10)	6730 (6)
C(35)	7767 (10)	8016 (10)	6603 (6)
C(36)	6750 (9)	7630 (9)	5866 (6)
C(61)	3624 (8)	7082 (8)	1545 (5)
C(62)	2840 (8)	7989 (9)	1853 (5)
C(63)	1661 (9)	7960 (9)	1329 (6)
C(64)	1259 (10)	7020 (10)	482 (7)
C(65)	2049 (9)	6143 (9)	161 (6)
C(66)	3240 (8)	6172 (9)	685 (5)
C(71)	6897 (8)	8217 (8)	1455 (5)
C(72)	8030 (9)	8108 (9)	1063 (6)
C(73)	8745 (9)	9236 (9)	817 (6)
C(74)	8343 (9)	10463 (9)	962 (6)
C(75)	7239 (10)	10576 (10)	1365 (7)
C(76)	6515 (9)	9463 (9)	1613 (6)
C(81)	5686 (8)	4582 (8)	1613 (5)
C(82)	6043 (9)	4451 (9)	2417 (5)
C(83)	5408 (9)	3189 (9)	2558 (6)
C(84)	4436 (10)	2056 (10)	1901 (6)
C(85)	4104 (10)	2202 (10)	1099 (6)
C(86)	4737 (9)	3448 (9)	946 (5)

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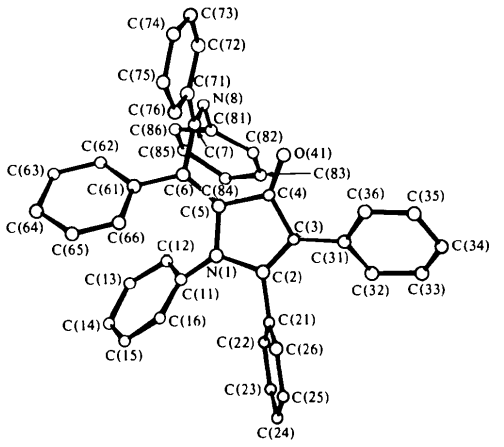

Fig. 1. A molecule of $C_{42}H_{30}N_2O$ showing the atom numbering.

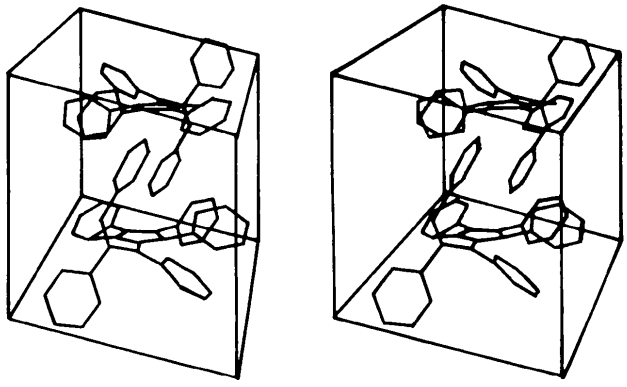

Fig. 2. Stereoscopic view of the molecules in the unit cell.

Table 2. *Bond lengths* (Å)

N(1)–C(11)	1·428 (5)	C(4)–C(5)	1·505 (9)
C(11)–C(12)	1·395 (7)	C(5)–N(1)	1·408 (10)
C(12)–C(13)	1·389 (7)	C(5)–C(6)	1·360 (10)
C(13)–C(14)	1·383 (7)	C(6)–C(61)	1·475 (7)
C(14)–C(15)	1·390 (7)	C(61)–C(62)	1·399 (7)
C(15)–C(16)	1·399 (7)	C(62)–C(63)	1·387 (7)
C(16)–C(11)	1·385 (7)	C(63)–C(64)	1·393 (7)
N(1)–C(2)	1·402 (9)	C(64)–C(65)	1·387 (7)
C(2)–C(21)	1·479 (7)	C(65)–C(66)	1·397 (7)
C(21)–C(22)	1·397 (7)	C(66)–C(61)	1·400 (7)
C(22)–C(23)	1·398 (7)	C(6)–C(7)	1·511 (10)
C(23)–C(24)	1·386 (7)	C(7)–C(71)	1·503 (7)
C(24)–C(25)	1·383 (7)	C(71)–C(72)	1·396 (7)
C(25)–C(26)	1·384 (7)	C(72)–C(73)	1·398 (7)
C(26)–C(21)	1·389 (7)	C(73)–C(74)	1·388 (7)
C(2)–C(3)	1·365 (10)	C(74)–C(75)	1·380 (7)
C(3)–C(31)	1·464 (7)	C(75)–C(76)	1·391 (7)
C(31)–C(32)	1·396 (7)	C(76)–C(71)	1·394 (7)
C(32)–C(33)	1·401 (7)	C(7)–N(8)	1·275 (9)
C(33)–C(34)	1·387 (7)	N(8)–C(81)	1·417 (6)
C(34)–C(35)	1·388 (7)	C(81)–C(82)	1·394 (7)
C(35)–C(36)	1·392 (7)	C(82)–C(83)	1·393 (7)
C(36)–C(31)	1·396 (7)	C(83)–C(84)	1·394 (7)
C(3)–C(4)	1·462 (10)	C(84)–C(85)	1·394 (7)
C(4)–O(41)	1·227 (8)	C(85)–C(86)	1·388 (7)
		C(86)–C(81)	1·393 (7)

Table 3. *Bond angles* (°)

C(11)–N(1)–C(2)	123·4 (5)	C(4)–C(5)–C(6)	124·7 (7)
C(11)–N(1)–C(5)	124·1 (5)	C(4)–C(5)–N(1)	106·5 (6)
C(2)–N(1)–C(5)	108·1 (6)	C(6)–C(5)–N(1)	128·6 (7)
C(16)–C(11)–C(12)	121·6 (7)	C(5)–C(6)–C(7)	119·1 (7)
N(1)–C(11)–C(12)	118·8 (7)	C(5)–C(6)–C(61)	124·9 (6)
N(1)–C(11)–C(16)	119·6 (7)	C(7)–C(6)–C(61)	116·0 (6)
C(11)–C(12)–C(13)	118·4 (8)	C(6)–C(61)–C(62)	120·3 (7)
C(12)–C(13)–C(14)	120·7 (8)	C(6)–C(61)–C(66)	120·3 (7)
C(13)–C(14)–C(15)	120·7 (8)	C(66)–C(61)–C(62)	119·3 (7)
C(14)–C(15)–C(16)	119·4 (8)	C(61)–C(62)–C(63)	121·0 (8)
C(15)–C(16)–C(11)	119·2 (8)	C(62)–C(63)–C(64)	119·4 (8)
N(1)–C(2)–C(3)	112·5 (6)	C(63)–C(64)–C(65)	120·2 (7)
N(1)–C(2)–C(21)	117·9 (6)	C(64)–C(65)–C(66)	120·6 (8)
C(21)–C(2)–C(3)	129·5 (6)	C(65)–C(66)–C(61)	119·4 (7)
C(2)–C(21)–C(22)	120·6 (7)	C(6)–C(7)–N(8)	123·6 (6)
C(2)–C(21)–C(26)	120·0 (7)	C(6)–C(7)–C(71)	117·7 (6)
C(22)–C(21)–C(26)	119·3 (7)	N(8)–C(7)–C(71)	118·3 (6)
C(21)–C(22)–C(23)	119·4 (7)	C(7)–C(71)–C(72)	119·2 (7)
C(22)–C(23)–C(24)	120·9 (8)	C(7)–C(71)–C(76)	121·3 (7)
C(23)–C(24)–C(25)	119·1 (9)	C(76)–C(71)–C(72)	119·5 (8)
C(24)–C(25)–C(26)	120·8 (8)	C(71)–C(72)–C(73)	119·6 (8)
C(25)–C(26)–C(21)	120·5 (8)	C(72)–C(73)–C(74)	120·7 (8)
C(2)–C(3)–C(4)	107·0 (6)	C(73)–C(74)–C(75)	119·5 (8)
C(2)–C(3)–C(31)	129·7 (6)	C(74)–C(75)–C(76)	120·6 (8)
C(31)–C(3)–C(4)	123·3 (6)	C(75)–C(76)–C(71)	120·2 (8)
C(3)–C(31)–C(32)	119·7 (7)	C(7)–N(8)–C(81)	121·9 (6)
C(3)–C(31)–C(36)	122·0 (7)	N(8)–C(81)–C(82)	120·9 (7)
C(36)–C(31)–C(32)	118·3 (6)	N(8)–C(81)–C(86)	118·2 (7)
C(31)–C(32)–C(33)	120·9 (6)	C(86)–C(81)–C(82)	120·5 (7)
C(32)–C(33)–C(34)	120·2 (8)	C(81)–C(82)–C(83)	119·4 (8)
C(33)–C(34)–C(35)	119·0 (8)	C(82)–C(83)–C(84)	120·6 (8)
C(34)–C(35)–C(36)	121·0 (8)	C(83)–C(84)–C(85)	119·1 (8)
C(35)–C(36)–C(31)	120·6 (8)	C(84)–C(85)–C(86)	121·0 (8)
C(3)–C(4)–C(5)	105·7 (6)	C(85)–C(86)–C(81)	119·3 (8)
C(3)–C(4)–O(41)	129·6 (7)		
C(5)–C(4)–O(41)	124·6 (7)		

technique on a Syntex automatic four-circle diffractometer with graphite-monochromated Cu $K\alpha$ radiation. Lp but not absorption corrections were applied. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971) and refined with *SHELX* (Sheldrick, 1976) by full-matrix least squares with all atoms anisotropic except H, for which a common isotropic temperature factor of 0·07 Å^2 was used. H atoms were placed in calculated positions and refined starting from these positions. Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Refinement converged with unit weights to $R = 0{\cdot}061$. A final ΔF synthesis was featureless. Final atomic parameters are given in Table 1,* bond lengths and angles in Tables 2 and 3 respectively.

* Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34618 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. The atom numbering is shown in Fig. 1 and a stereoscopic view of the molecule in Fig. 2.

N(1), C(2), C(3), C(4) and C(5) of the inner ring are situated in a plane [$0{\cdot}240x - 0{\cdot}955y - 0{\cdot}175z + 5{\cdot}549 = 0$ (Å)] from which C(2) has the greatest deviation [0·022 (8) Å].

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References

CHATILA, G. (1976). Diplomarbeit, Univ. Hamburg.
EICHER, TH. & WEBER, J. L. (1975). *Top. Curr. Chem./Fortschr. Chem. Forsch.* **57**, 1–109.
GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
SHELDRICK, G. M. (1976). *SHELX*. A program for crystal structure determination. Univ. of Cambridge, England.

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Triméthyl-3,3,5 Phényl-5 Cyclohexanone-oxime

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Abstract. $C_{15}H_{21}NO$, $M_r = 231{\cdot}3$, monoclinic, $P2_1/c$, $a = 14{\cdot}98$ (2), $b = 7{\cdot}47$ (1), $c = 12{\cdot}41$ (2) Å, $\beta = 105{\cdot}5$ (2)°, $Z = 4$, $D_c = 1{\cdot}15$ Mg m^{-3}. The final R was 0·083. The molecule possesses a chair-type conformation, with the phenyl in an axial position. There are strong interactions between the axial methyl and phenyl groups.

Introduction. La structure géométrique de la cétone correspondante a fait l'objet d'une longue controverse, concernant d'une part sa conformation stable, d'autre part l'orientation du groupe phényle par rapport au cycle. La cétone étant liquide à la température ambiante, nous avons préparé et recristallisé son oxime; une étude préalable nous ayant montré qu'au cours de la réaction il se formait les deux isomères *syn* et *anti* de l'oxime, nous avons recristallisé notre produit jusqu'à l'obtention d'un cristal homogène en l'isomère le plus abondant. Une étude préliminaire a été effectuée sur chambre de Weissenberg. Les paramètres de la maille ont été affinés à partir des mesures exactes des conditions géométriques de diffraction de 15 réflexions (diffractomètre automatique quatre cercles CAD-4 Enraf–Nonius; radiation Cu $K\alpha$). 2275 réflexions ont été mesurées ($\theta \leq 65°$), dont 1299 mesurées avec suffisamment de précision ont été conservées [$I \geq 3\sigma(I)$].

Nous avons effectué les corrections de Lorentz–polarisation. Nous n'avons pas tenu compte des effets d'absorption étant donné les faibles dimensions du cristal (0,2 × 0,15 × 0,45 mm environ), et la valeur peu élevée du coefficient d'absorption linéaire ($\mu = 0{,}56$ mm^{-1}).

La structure a été déterminée par l'utilisation des méthodes directes, à l'aide du programme *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974).

La synthèse de Fourier obtenue avec les facteurs de structure normalisés E affectés des phases donnant la meilleure figure de mérite, a permis de placer tous les atomes, sauf les hydrogènes.

Nous avons ensuite effectué un affinement par moindres carrés à l'aide du programme de Busing, Martin & Levy (1962). Par application de la méthode de Hughes (1941), nous avons pris comme poids la quantité $W = 1/(A + BF_o^2)$, avec $A = 1{,}25$, $B = 0{,}002$, afin que la quantité $\overline{W\Delta F}$ soit indépendante de F_o. Nous avons pris une agitation thermique anisotrope

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