

Table 2. Bond lengths (Å) and angles (°)

N(1)—C(2)	1.332 (2)	O(9)—C(15)	1.391 (3)
C(2)—C(3)	1.400 (3)	C(10)—C(11)	1.395 (3)
C(2)—C(10)	1.444 (3)	C(11)—C(12)	1.383 (3)
C(3)—C(4)	1.374 (3)	C(12)—C(13)	1.386 (4)
C(3)—O(9)	1.367 (2)	C(13)—C(14)	1.387 (4)
C(4)—C(5)	1.376 (3)	C(14)—C(15)	1.375 (3)
C(5)—C(6)	1.403 (3)	C(15)—C(10)	1.392 (3)
C(6)—N(1)	1.347 (3)		
C(6)—C(7)	1.437 (3)		
C(7)—N(8)	1.141 (3)		
N(1)—C(2)—C(3)	123.1 (1)	O(9)—C(15)—C(14)	124.0 (1)
N(1)—C(2)—C(10)	130.8 (1)	O(9)—C(15)—C(10)	111.6 (1)
C(2)—C(3)—C(4)	122.5 (1)	C(10)—C(2)—C(3)	106.0 (1)
C(2)—C(3)—O(9)	111.4 (1)	C(10)—C(11)—C(12)	117.8 (2)
C(3)—C(4)—C(5)	114.8 (2)	C(11)—C(12)—C(13)	121.8 (2)
C(4)—C(5)—C(6)	119.9 (2)	C(12)—C(13)—C(14)	121.5 (2)
C(5)—C(6)—N(1)	125.1 (1)	C(13)—C(14)—C(15)	115.7 (2)
C(5)—C(6)—C(7)	119.3 (2)	C(14)—C(15)—C(10)	124.2 (2)
C(6)—C(7)—N(8)	178.7 (3)	C(15)—C(10)—C(11)	118.8 (1)
C(6)—N(1)—C(2)	114.3 (1)		
C(7)—C(6)—N(1)	115.5 (2)		
O(9)—C(3)—C(4)	126.0 (1)		

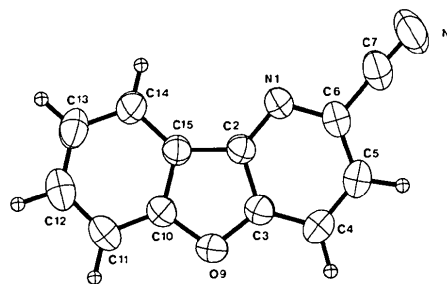


Fig. 1. Thermal-ellipsoid plot (Johnson, 1965). Ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

References

- ITOH, T., OHSAWA, A., ITOH, Y. & IGETA, H. (1990). *Heterocycles*. In the press.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Rigaku Corporation (1985). *RCRYSTAN. X-ray Analysis Program System*. Rigaku Corporation, Tokyo, Japan.
- YAO, J.-X., ZHENG, C.-D., QIAN, J.-Z., HAN, F.-S., GU, Y.-X. & FAN, H.-F. (1985). *SAPR85. A Computer Program for the Automatic Solution of Crystal Structure from X-ray Diffraction Data*. Institute of Physics, Academia Sinica, Beijing, People's Republic of China.

Fig. 1 shows a thermal-ellipsoid plot of the molecule with atomic labeling.

Related literature. The title compound was obtained by flash vacuum pyrolysis of 2-cyano-6-phenylpyridine *N*-oxide (Itoh, Ohsawa, Itoh & Igeta, 1990).

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Structure of 3-(1-Aziridinyl)-*N*-(*p*-chlorophenyl)succinimide*

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Abstract. C₁₂H₁₁ClN₂O₂, *M_r* = 250.7, monoclinic, *P*2₁/*a*, *a* = 8.328 (4), *b* = 11.017 (5), *c* = 12.763 (2) Å, β = 93.99 (4)°, *V* = 1168 (1) Å³, *Z* = 4, *D_x* =

1.43 Mg m⁻³, λ(Mo Kα) = 0.7107 Å, μ = 0.314 mm⁻¹, *F*(000) = 520, *T* = 293 K, *R* = 0.045 for 1267 observed reflections. The X-ray analysis establishes unequivocally the molecular structure of the title compound. The aziridinyl ring is planar. The succinimide ring is a β-envelope with C(2) as the flap

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at $-0.324(3)$ Å from the best plane through the other four ring atoms. The Δ and φ_m values [Altona, Geise & Romers (1968). *Tetrahedron*, **24**, 13–32] are $21.4(3)$, $21.0(3)^\circ$, respectively. The dihedral angle between the aziridinyll and succinimide rings is only $5.4(3)^\circ$. The *p*-chlorophenyl ring is planar and rotated by $143.8(1)^\circ$ from the mean plane of the five-membered ring. The phenyl ring shows normal geometry with mean bond lengths and angles of $1.372(4)$ Å and $120.1(3)^\circ$. Four intermolecular contacts < 3.42 Å are present: C(3)—H...N(2) ($-x, -y, -z$) $3.262(4)$, C(6)—H...Cl($x, y, 1+z$) $3.408(3)$, C(2)—H...O(1) ($\frac{1}{2}+x, \frac{1}{2}-y, z$) $3.358(4)$ and C(8)—H...O(2) ($-\frac{1}{2}+x, \frac{1}{2}-y, z$) $3.416(4)$ Å.

Experimental. Colourless crystal, $0.40 \times 0.40 \times 0.30$ mm. Nicolet R3m four-circle diffractometer, graphite-monochromated Mo K α radiation. Lattice parameters from 25 machine-centred reflections with $4.9 < 2\theta < 18.5^\circ$. 1521 reflections with $3 < 2\theta < 45^\circ$ for one octant, 1267 independent with $I > 3.0\sigma(I)$. Index range h 0→9, k 0→12, l 0→13, ω -scan mode, variable scan speed. Two standard reflections (131, 220) monitored every 50 measurements; no significant variation. Lp correction. Data adjusted to an approximately absolute scale and an overall U value of 0.049 Å², absorption ignored and $R_{\text{int}} = 0.028$. Structure solved by direct methods and partial structure expansion by an iterative *E*-Fourier procedure using *SHELXTL5* (Sheldrick, 1985). Least-squares refinement with all non-H atoms anisotropic; H atoms of CH and CH₂ were allowed to ride on bonded C atoms with a fixed isotropic $U = 0.06$ Å². $\sum w(\Delta F)^2$ minimized, $w[\sigma^2(F_o) + 0.002(F_o)^2]^{-1}$, where σ is the standard deviation of the observed amplitudes, based on counting statistics. In the last cycle $(\Delta/\sigma)_{\text{max}} = 0.107$; $\Delta\rho$ from -0.27 to 0.32 e Å⁻³; isotropic extinction parameter $X = 0.0059$, $S = 1.19$; final $R = 0.045$, $wR = 0.066$; scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a Nova 4S computer and plots drawn on a Tektronix plotter with the *SHELXTL* system of programs.

Atomic coordinates are in Table 1. A perspective molecular drawing and the atom labelling are displayed in Fig. 1. Bond distances and angles are listed in Table 2.*

Related literature. The title compound was prepared as part of our investigation of the reaction of maleimides and isomaleimides with aziridine (Joseph-Nathan, Mendoza & Garcia, 1972, 1974). Its struc-

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53018 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors (Å² $\times 10^3$)

	x	y	z	U_{eq}
Cl	781 (2)	3259 (1)	-5849 (1)	104 (1)
O(1)	-190 (3)	2316 (2)	-765 (2)	57 (1)
O(2)	3826 (3)	-118 (2)	-1816 (2)	57 (1)
N(1)	1793 (2)	1253 (2)	-1549 (2)	37 (1)
N(2)	726 (3)	805 (2)	1075 (2)	42 (1)
C(1)	967 (3)	1656 (3)	-703 (2)	40 (1)
C(2)	1823 (3)	1156 (3)	282 (2)	43 (1)
C(3)	2786 (3)	118 (3)	-97 (2)	50 (1)
C(4)	2928 (3)	367 (3)	-1238 (2)	43 (1)
C(5)	1493 (4)	798 (3)	2143 (2)	58 (1)
C(6)	366 (4)	1754 (3)	1815 (2)	58 (1)
C(7)	1548 (3)	1721 (2)	-2595 (2)	39 (1)
C(8)	1281 (4)	2942 (3)	-2746 (2)	53 (1)
C(9)	1037 (4)	3409 (3)	-3737 (2)	65 (1)
C(10)	1070 (4)	2652 (3)	-4584 (3)	62 (1)
C(11)	1329 (4)	1435 (3)	-4451 (3)	65 (1)
C(12)	1564 (4)	960 (3)	-3454 (2)	52 (1)

Table 2. Bond lengths (Å) and angles ($^\circ$) with e.s.d.'s in parentheses

Cl—C(10)	1.748 (3)	O(1)—C(1)	1.205 (3)
O(2)—C(4)	1.211 (4)	N(1)—C(1)	1.392 (3)
N(1)—C(4)	1.396 (3)	N(1)—C(7)	1.433 (3)
N(2)—C(2)	1.461 (4)	N(2)—C(5)	1.464 (4)
N(2)—C(6)	1.454 (4)	C(1)—C(2)	1.506 (4)
C(2)—C(3)	1.496 (4)	C(3)—C(4)	1.494 (4)
C(5)—C(6)	1.452 (5)	C(7)—C(8)	1.375 (4)
C(7)—C(12)	1.380 (4)	C(8)—C(9)	1.367 (4)
C(9)—C(10)	1.367 (5)	C(10)—C(11)	1.367 (5)
C(11)—C(12)	1.378 (4)		
C(1)—N(1)—C(4)	111.3 (2)	C(1)—N(1)—C(7)	124.2 (2)
C(4)—N(1)—C(7)	124.4 (2)	C(2)—N(2)—C(5)	113.1 (2)
C(2)—N(2)—C(6)	115.3 (2)	C(5)—N(2)—C(6)	59.7 (2)
O(1)—C(1)—N(1)	125.2 (2)	O(1)—C(1)—C(2)	127.3 (3)
N(1)—C(1)—C(2)	107.5 (2)	N(2)—C(2)—C(1)	113.1 (2)
N(2)—C(2)—C(3)	113.4 (2)	C(1)—C(2)—C(3)	104.0 (2)
C(2)—C(3)—C(4)	104.8 (2)	O(2)—C(4)—N(1)	124.7 (3)
O(2)—C(4)—C(3)	127.3 (3)	N(1)—C(4)—C(3)	107.9 (2)
N(2)—C(5)—C(6)	59.8 (2)	N(2)—C(6)—C(5)	60.5 (2)
N(1)—C(7)—C(8)	119.5 (2)	N(1)—C(7)—C(12)	120.9 (2)
C(8)—C(7)—C(12)	119.6 (3)	C(7)—C(8)—C(9)	120.6 (3)
C(8)—C(9)—C(10)	119.5 (3)	Cl—C(10)—C(9)	119.2 (3)
Cl—C(10)—C(11)	120.0 (3)	C(9)—C(10)—C(11)	120.8 (3)
C(10)—C(11)—C(12)	119.9 (3)	C(7)—C(12)—C(11)	119.6 (3)

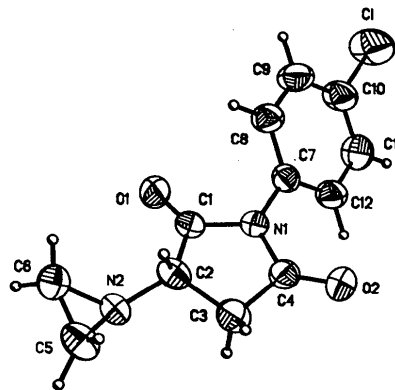


Fig. 1. The molecular structure of the title compound with numbering.

ture was investigated by NMR and could not be established unambiguously.

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References

ALTONA, C., GEISE, H. J. & ROMERS, C. (1968). *Tetrahedron*, **24**, 13–32.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOSEPH-NATHAN, P., MENDOZA, V. & GARCIA G., E. (1972). *J. Org. Chem.* **37**, 3950–3952.

JOSEPH-NATHAN, P., MENDOZA, V. & GARCIA G., E. (1974). *Can. J. Chem.* **52**, 129–131.

SHELDRIK, G. M. (1985). *SHELXTL5. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Federal Republic of Germany.

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Structure of Kostanecki's Triketone

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Abstract. 2,4-Dibenzoyl-1,3,5-triphenylcyclohexanol, $C_{38}H_{32}O_3$, $M_r = 536.7$, monoclinic, $C2/c$, $a = 41.651$ (13), $b = 6.299$ (2), $c = 23.638$ (7) Å, $\beta = 114.17$ (2)°, $V = 5658.0$ Å³, $Z = 8$, $D_x = 1.260$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.851$ cm⁻¹, $F(000) = 2272$, $T = 153$ K. Final $R = 0.046$, $wR = 0.055$ for 2185 unique observed reflections. All the bulky side groups are in equatorial positions and the single hydroxyl group forms an intramolecular hydrogen bond to the O atom of a neighbouring benzoyl group with an O...O distance of 2.717 (7) Å.

Introduction. The title compound is one of the satellite products of the Mannich reaction to synthesize α -aminomethyl, α' -methylene-1,5-diketones (Pavel & Tilichenko, 1973). It was identified as the low-temperature fusion form of Kostanecki's triketone (Kostanecki & Rossbach, 1896). The present X-ray diffractometric investigation was undertaken to determine the molecular structure, and to define its stereochemistry.

Experimental. Colourless crystals were obtained by slow evaporation of a solution in ethylmethyl ketone. A crystal approximately $0.2 \times 0.2 \times 0.3$ mm was used for the measurements. D_m not measured. Mo $K\alpha$ radiation was used with a graphite-crystal monochromator on a Syntex $P2_1$ single-crystal diffractometer. The unit-cell dimensions were

determined from the angular settings of 24 reflections ($15 \leq \theta \leq 40^\circ$) at 153 K. The space group was determined to be $C2/c$ from systematic absences.

The intensity data reflections (half of the sphere up to $2\theta = 56^\circ$, $0 \leq h \leq 51$, $0 \leq k \leq 9$, $-29 \leq l \leq 29$) were measured using the $\omega/2\theta$ -scan technique and a variable scan rate with a maximum of $30^\circ \text{ min}^{-1}$. The intensity of the primary beam was checked throughout the data collection by monitoring three reference reflections after every 200. Space-group symmetry-equivalent reflections were averaged, resulting in 3931 unique reflections of which 2184 were observed with $I > 6\sigma(I)$. Lorentz and polarization corrections were applied, and the data were reduced to $|F_o|$ values.

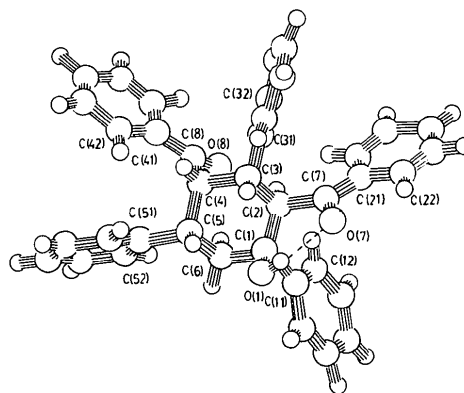


Fig. 1. Molecular structure and atom-numbering scheme.