To adapt itself to the steric strain caused by the presence of substituents, the 8,9,10-trinorbornane framework assumes the synchro-twist $S(-,-)$ conformation, as follows from comparison of the absolute values of the torsion angles (Altona & Sundaralingam, 1970): |C(7)—C(1)—C(2)—C(3)| [31·2 (3)°] < |C(7)—C(4)—C(3)—C(2)| [37·2 (3)°]; |C(7)—C(1)—C(6)—C(5)| [39·0 (3)°] > |C(7)—C(4)—C(5)—C(6)| [31·4 (3)°]. The C(1)C(2)C(3)C(4)C(7) five-membered ring is distorted to a somewhat smaller extent than the C(1)C(6)C(5)C(4)C(7) ring, the total puckering amplitudes Q being 0·570 (3) and 0·599 (3) Å, respectively.

Substituted bicyclo[2.2.1]heptane derivatives with 2-nitrophenylthio substituents often show a shortening of the S···O(nitro group) non-bonded distance in comparison with the sum of their van der Waals radii, 3·25 Å (Pauling, 1960). Thus in the structures of 3-*exo*-methoxy-5-*exo*-(2-nitrophenylthio)tricyclo-[2.2.1.0^{2,6}]heptane (Garratt, Przybylska & Cygler, 1983), 1-chloromethyl-3-*endo*-(2,4-dinitrophenylthio)-tricyclo[2.2.1.0^{2,0}]heptane (Przybylska & Garratt, 1981) and 2-*exo*-chloro-7,7-dimethyl-3-*endo*-(2-nitrophenylthio)bicyclo[2.2.1]heptane (Sergeyeva, Struchkov, Kurkutova, Zemlyanukhina, Sadovaya & Zefirov, 1984) these distances are respectively 2·694 (2), 2·739 (2) and 2·694 (2) Å. Their shortening can be considered as the result of the attractive interaction between the S and O atoms. In molecule (IV) the S···O(5) distance is somewhat longer and the *ortho* nitro group is more significantly rotated out of the benzene plane [the dihedral angle in (IV) is equal to 40·6°, whereas in the structures mentioned above these angles are respectively 25·0, 28·0 and 11·9°]. In molecule (IV), along with the considerably shortened S···O(5) distance, the less significantly shortened non-bonded distance S···O(1) 2·987 (2) Å is also observed. The directions of both shortened contacts are close to the C—S bond vectors [the C(7)—S···O(5) and C(8)—S···O(1) angles are 162·1 (1) and 172·8 (1)°, respectively], *i.e.* the T-type coordination of the S atom, characteristic of molecules with shortened S–nucleophile contacts, is observed.

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1-(4-Chlorophenyl)-2-cyclopentylethanone

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Abstract. $C_{13}H_{15}ClO$, $M_r = 222·72$, monoclinic, $P2_1/a$, $a = 7·936$ (2), $b = 10·741$ (3), $c = 14·001$ (4) Å, $\beta = 101·52$ (1)°, $V = 1169·4$ (5) Å^3, $Z = 4$, $D_x = 1·265$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 2·96$ cm^{-1}, $F(000) = 472$, $T = 295$ K, final $R = 0·061$ for 1342 observed reflections. The carbonyl-containing side chain occupies an equatorial position with respect to the five-membered cyclopentane ring; the conformation of the ring is close to C_2 (half-chair or twisted form). The equatorial γ-H atom is 2·76 (4) Å from the carbonyl O atom [axial H 3·46 (4) Å], which is near the upper limit for H abstraction.

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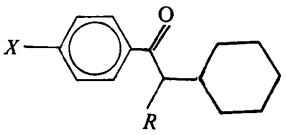
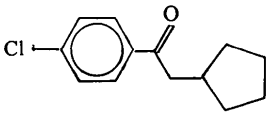
Introduction. Several structural analyses on α-cyclohexylacetophenones (1) have been reported (Ariel & Trotter, 1985, 1986*a,b*) in conjunction with photochemical studies of their solid-state reactions, *i.e.* the Norrish type II reaction with γ-H abstraction by the carbonyl O atom leading to cleavage and cyclization (Ariel, Ramamurthy, Scheffer & Trotter, 1983). As part of these continuing studies, a structural determination of the present compound (2) was undertaken to verify the molecular conformation in the solid state and to elucidate the effect of the size of the ring on the solid-state photoreaction.



(1)



(2)

(1*a*) $R = H$; $X = CH_3$, Cl, CH_3O, COOH, CN
(1*b*) $R = CH_3$; $X = COOH$
(1*c*) $R = C_6H_5$; $X = CH_3O$

Experimental. Crystal size 0·1 × 0·3 × 0·5 mm, m.p. 333–334 K, Enraf–Nonius CAD-4F single-crystal X-ray diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, lattice parameters from setting of 25 reflections with $17 \leq \theta \leq 21°$. 2124 unique reflections with $\theta \leq 27{\cdot}5°$; $h = -10$–10, $k = 0$–14, $l = 0$–18; ω–2θ scan, ω scan width $(1{\cdot}0 + 0{\cdot}35\tan\theta)°$, extended 25% on each side for background measurement, horizontal aperture $(1{\cdot}0 + \tan\theta)$ mm, vertical aperture 4 mm; Lp corrections, no absorption correction; three standard reflections, 10% decay (correction applied). Structure solved by direct methods with use of *SHELX* (Sheldrick, 1976) and refined by full-matrix least squares with minimization of $\sum w(|F_o| - |F_c|)^2$. All H atoms located in difference Fourier synthesis and refined isotropically. 196 parameters, consisting of 90 positional parameters, 90 anisotropic thermal parameters, 15 isotropic thermal parameters, and a scale factor. Final $R = 0{\cdot}061$, $wR = 0{\cdot}045$ for 1342 reflections for which $F \geq 3\sigma(F)$, where $\sigma^2(I) = S + 2B + [0{\cdot}04(S - B)]^2$, S = scan count, B = time-averaged background count; four strong reflections given zero weight during refinement because of extinction. $R = 0{\cdot}114$, $wR = 0{\cdot}059$ for all data, $w = 1/\sigma^2(F)$, $(\Delta/\sigma)_{max} = 0{\cdot}02$, $\pm 0{\cdot}28$ e Å^{-3} in final difference synthesis; atomic scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). All calculations performed on a 16 Mbyte Amdahl 470 V/8 (MTS-G) computer. Diagrams drawn with *PLUTO* (Motherwell & Clegg, 1978).

Table 1. *Atom coordinates* ($\times 10^4$ *for* Cl, O, C; $\times 10^3$ *for* H) *and temperature factors* (Å$^2 \times 10^3$)

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

	x	y	z	U_{eq}†
Cl(1)	2241 (1)	4043 (1)	1865 (<1)	84
O(1)	447 (3)	2621 (2)	6188 (1)	70
C(1)	858 (4)	2790 (3)	4265 (2)	53
C(2)	1059 (4)	2904 (3)	3325 (2)	61
C(3)	1962 (4)	3901 (3)	3060 (2)	56
C(4)	2632 (4)	4797 (3)	3727 (2)	58
C(5)	2436 (4)	4663 (3)	4677 (2)	55
C(6)	1560 (3)	3663 (2)	4970 (2)	46
C(7)	1328 (3)	3474 (3)	5993 (2)	52
C(8)	2203 (5)	4358 (3)	6770 (2)	55
C(9)	1914 (4)	4118 (3)	7786 (2)	56
C(10)	2794 (6)	2974 (4)	8296 (2)	76
C(11)	3002 (8)	3235 (5)	9384 (3)	103
C(12)	2554 (7)	4578 (4)	9491 (3)	90
C(13)	2552 (5)	5164 (3)	8511 (2)	71
H(1)	20 (3)	219 (3)	449 (2)	56 (8)
H(2)	65 (3)	237 (2)	287 (2)	62 (9)
H(4)	322 (4)	552 (3)	352 (2)	81 (9)
H(5)	288 (3)	526 (2)	514 (2)	56 (8)
H(81)	185 (3)	516 (3)	657 (2)	53 (8)
H(82)	341 (4)	433 (2)	677 (2)	54 (8)
H(9)	73 (3)	407 (2)	776 (2)	45 (7)
H(101)	227 (4)	224 (3)	806 (2)	86 (12)
H(102)	395 (5)	290 (3)	810 (2)	120 (14)
H(111)	234 (6)	265 (4)	966 (3)	152 (19)
H(112)	414 (6)	307 (4)	966 (3)	141 (19)
H(121)	135 (6)	461 (4)	962 (3)	151 (17)
H(122)	321 (4)	496 (3)	1003 (2)	80 (10)
H(131)	377 (4)	533 (3)	846 (2)	75 (9)
H(132)	176 (4)	590 (3)	837 (2)	86 (10)

† Isotropic temperature factors for H atoms.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

C(1)–C(2)	1·363 (4)	C(7)–C(8)	1·506 (4)
C(2)–C(3)	1·380 (4)	C(8)–C(9)	1·508 (4)
C(3)–C(4)	1·372 (4)	C(9)–C(10)	1·519 (4)
C(4)–C(5)	1·377 (4)	C(10)–C(11)	1·525 (5)
C(5)–C(6)	1·385 (3)	C(11)–C(12)	1·501 (6)
C(1)–C(6)	1·395 (3)	C(12)–C(13)	1·510 (5)
C(6)–C(7)	1·493 (3)	C(9)–C(13)	1·531 (4)
C(7)–O(1)	1·216 (3)	C(3)–Cl(1)	1·738 (3)
C(6)–C(1)–C(2)	121·1 (3)	C(8)–C(7)–O(1)	121·0 (3)
C(3)–C(2)–C(1)	119·8 (3)	C(8)–C(7)–C(6)	118·6 (2)
C(4)–C(3)–C(2)	120·9 (3)	C(9)–C(8)–C(7)	116·0 (3)
C(2)–C(3)–Cl(1)	120·0 (2)	C(10)–C(9)–C(8)	116·1 (3)
C(4)–C(3)–Cl(1)	119·2 (2)	C(13)–C(9)–C(8)	114·0 (3)
C(5)–C(4)–C(3)	118·7 (3)	C(13)–C(9)–C(10)	102·8 (3)
C(6)–C(5)–C(4)	121·9 (3)	C(11)–C(10)–C(9)	105·6 (3)
C(5)–C(6)–C(1)	117·6 (3)	C(12)–C(11)–C(10)	107·2 (3)
C(7)–C(6)–C(1)	118·6 (2)	C(13)–C(12)–C(11)	105·5 (3)
C(7)–C(6)–C(5)	123·8 (2)	C(12)–C(13)–C(9)	103·7 (3)
C(6)–C(7)–O(1)	120·3 (2)		

		C_s*	C_2*
C(10)–C(11)–C(12)–C(13)	−14·6 (5)	0·0	−13·2
C(11)–C(12)–C(13)–C(9)	33·4 (4)	25·0	34·3
C(12)–C(13)–C(9)–C(10)	−39·2 (4)	−40·3	−42·3
C(13)–C(9)–C(10)–C(11)	30·2 (4)	40·3	34·3
C(9)–C(10)–C(11)–C(12)	−10·0 (5)	−25·0	−13·2
C(1)–C(6)–C(7)–O(1)	−4·9 (4)		
C(1)–C(6)–C(7)–C(8)	175·2 (3)		
C(5)–C(6)–C(7)–O(1)	175·1 (3)		
C(5)–C(6)–C(7)–C(8)	−4·8 (4)		
O(1)–C(7)–C(8)–C(9)	0·0 (4)		
C(6)–C(7)–C(8)–C(9)	179·9 (2)		
C(7)–C(8)–C(9)–C(10)	71·1 (4)		
C(7)–C(8)–C(9)–C(13)	−169·6 (3)		
C(8)–C(9)–C(10)–C(11)	155·3 (3)		

* Calculated: Adams, Geise & Bartell (1970).

Discussion. Final atomic coordinates are in Table 1, bond distances, bond angles and selected torsion angles in Table 2.* A stereoview of the molecular conformation is in Fig. 1. The C(sp^3)–C(sp^3) bond distances (Table 2) are in the range 1·501 (6)–1·531 (4) Å, mean 1·516 (9) Å. The cyclopentane ring angles are in the range 102·8 (3)–107·2 (3)°, mean 105 (1)°.

A cyclopentane ring may exhibit envelope (C_s) or half-chair (C_2) conformations, which are about 21 kJ mol^{-1} more stable than the planar arrangement (Carreira, Jiang, Person & Willis, 1972), with interconversion between the two stable conformations *via* pseudorotation in the gas phase (Adams, Geise & Bartell, 1970) or even in the solid state (Margulis, Dalton & Kwiram, 1973). Torsion angles for the present structure (Table 2) indicate a conformation close to C_2. As for pseudorotation, the cyclopentane ring appears to be rigid, showing no evidence of disorder, although the γ-C atoms [C(11), C(12)] have higher thermal parameters than the other atoms in the molecule (see Table 1). The value of q, calculated as the square root of the sum of squares of the deviations of the atoms from the mean plane through the five-membered ring (Dunitz, 1979) is 0·38 Å. Adams *et al.* (1970) determined q to be 0·44 Å, while other estimates, listed by Adams, have yielded values as high as 0·48 Å.

Compound (2) crystallizes in a similar conformation to the seven derivatives of (1), in that the carbonyl-containing side chain is equatorial-like with respect to the cyclopentane ring (Ariel & Trotter, 1985, 1986*a*,*b*). The molecular conformation of (2), as indicated by the torsion angles about the C(7)–C(8) bond, is very similar to that of compounds (1*a*) (Table 3); compounds (1*b*) and (1*c*) show rotations of 100 and 30° about C(7)–C(8) from the conformations of (1*a*) and (2). The equatorial γ-H atom, H(101), in (2) is closer to O(1) [2·76 (4) Å, compared with the range of 2·60–2·73 Å in compounds (1), Table 3] than the axial γ-H atom, H(102) [3·46 (4) Å], although the 2·76 Å distance is close to the suggested upper limit of 2·72 Å (van der Waals radii sum) for H abstraction (Appel, Jiang, Scheffer & Walsh, 1983). The corresponding τ and Δ angles are 31 and 96° for (2), compared with ranges of 42–65 and 73–91° for (1) (Table 3). The relative geometry of H(9) in compound (2) is similar to that of (1*a*) and (1*c*); here O(1)···H(9) is 2·66 (3) Å, $\tau = 15°$ and $\Delta = 78°$.

Solid-state and solution photochemistry of compound (2) results in the cleavage product, with no cyclization product being found. The presence of the cyclopentyl ring thus results in significantly different photochemical behaviour in comparison with the cyclohexyl derivatives, possibly as a result of strain which would exist in a possible photoproduct with fused four- and five-membered rings.

Intermolecular distances correspond to van der Waals interactions.

* Lists of structure factors, anisotropic thermal parameters and bond distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42931 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

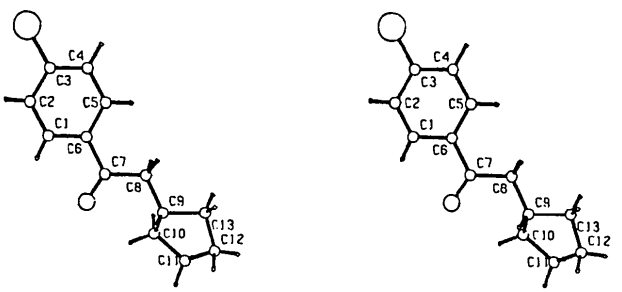


Fig. 1. Stereoscopic view of (2) with crystallographic atomic labelling.

Table 3. *Torsion angles* (°) *defining the molecular conformations, and solid-state* H-*abstraction distances* (Å) *and angles* (°)

The angle τ is the degree by which the H atom in question lies outside the mean plane of the carbonyl group. The angle Δ is formed between the H atom in question and the C(7)–O(1) bond.

	(1*a*)					(1*b*)	(1*c*)	(2)
	CH_3	Cl	CH_3O	COOH	CN			
Ketone/ring angle	13	4	13	11	3	21	12	5
C(6)–C(7)–C(8)–C(9)	167	−174	−173	172	178	74	140	180
O(1)–C(7)–C(8)–C(9)	−13	7	8	−10	−4	−103	−41	0
O···H_e	2·60	2·60	2·61	2·60	2·65	2·73	2·70	2·76
τ_e	50	42	43	44	42	65	63	31
Δ_e	88	90	91	90	88	73	76	96
O···H_α	3·83	3·83	3·82	3·81	3·90	3·54	3·90	3·46
τ_α	38	36	37	38	33	39	43	36
Δ_α	65	67	68	67	67	75	54	73
O···H_β	2·57	2·59	2·64	2·61	2·55	3·55	2·57	2·66
τ_β	7	13	12	10	13	28	13	15
Δ_β	84	81	81	82	82	30	84	78

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Acta Cryst. (1986). C**42**, 1169–1172

Structures of Radical Anion $TCNQ^{\cdot -}$ Salts with 1,2-Dithiole Derivatives. II. 3-*p*-Methoxyphenyl-1,2-dithiolylium—7,7,8,8-Tetracyano-*p*-quinodimethanide (1/1·5)

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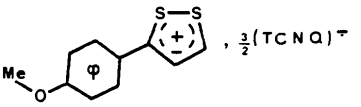
Abstract. $C_{10}H_9OS_2^+\cdot\frac{3}{2}C_{12}H_4N_4^{\cdot -}$, $M_r = 515\cdot6$, triclinic, $P\bar{1}$, $a = 7\cdot836$ (1), $b = 16\cdot316$ (2), $c = 10\cdot050$ (1) Å, $\alpha = 90\cdot3$ (1), $\beta = 71\cdot0$ (1), $\gamma = 87\cdot6$ (1)°, $V = 1213\cdot6$ (5) Å^3, $Z = 2$, $D_m = 1\cdot40$, $D_x = 1\cdot411$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71093$ Å, $\mu = 0\cdot253$ mm^{-1}, $F(000) = 530$, $T = 293$ K, $R = 0\cdot042$ and $wR = 0\cdot045$ for 2878 reflections. The TCNQ and 3-*p*-methoxyphenyl-1,2-dithiolylium ions form segregated stacks along the crystallographic *c* and *a* axes respectively. The stacking of the TCNQ ions is triadic with a spacing of 3·175 Å between the $TCNQ^{\cdot -}$ of a triad and 3·43 Å between triads. The material is expected to be an insulator along the stacking direction of the $TCNQ^{\cdot -}$.

Introduction. For more than ten years, many investigations have been undertaken in the field of organic conductors, particularly since the studies on the charge-transfer complex TTF–TCNQ (Ferraris, Cowan, Walatka & Perlstein, 1973; Phillips, Kistenmacher, Ferraris & Cowan, 1973; Kistenmacher, Phillips & Cowan, 1974), which exhibits a metallic state (Coleman, Cohen, Sandman, Yamagishi, Garito & Heeger, 1973). Besides the theoretical investigations carried out on this material and the models proposed to explain the behavior of organic conductors (Bardeen, 1973; Soos, 1974; Perlstein, 1977; Torrance & Silverman, 1977; Soos, 1978; Benoit, Galtier & Montaner, 1979), numerous charge-transfer complexes have been synthesized and investigated from derivatives of TTF–TCNQ (Delhaes, Keryer, Gaultier, Fabre & Giral, 1982).

In order to contribute to this new field of solid-state chemistry, Amzil, Le Coustumer & Mollier (1981) prepared a series of TCNQ salts with 1,2-dithiole derivatives as electron donors. In these derivatives, the dithiole ring is planar and carries a sextet of π electrons which gives it aromatic character. We have already studied the structure of a complex salt of 1:2 stoichiometry (Mathieu, 1984) which shows semiconductor behavior with respect to temperature. In the present work we study the structure of a salt of the same series but with a 2:3 stoichiometry, 3-*p*-methoxyphenyl-1,2-dithiolylium sesqui(7,7,8,8-tetracyano-*p*-quinodimethanide), of formula



Experimental. Very absorbent grey-blue crystals (in acetonitrile by evaporation and cooling); D_m by immersion in toluene–carbon tetrachloride (5:13·4); 0·20 × 0·30 × 0·30 mm; automatic diffractometer CAD-4 Enraf–Nonius; Mo $K\alpha$ with graphite-plate monochromator, Bragg angle max. $\theta_m = 32°$, ω–2θ scan, amplitude $\Delta = (0\cdot95 + 0\cdot35 \tan\theta)°$, counter aperture $(3\cdot00 + 0\cdot80 \tan)$ mm, scan speed $\sigma(I)/I = 0\cdot018$ for max. counting time 60 s; 25 reflections for redetermination of cell parameters; 2878 independent reflections with $I \geq 3\sigma(I)$; Lorentz–polarization corrections; $-9 \leq h \leq 11$, $-24 \leq k \leq 24$, $0 \leq l \leq 11$; 3 standard reflections, intensity fluctuations <3%; heavy-atom method; atomic scattering factors from Cromer & Waber (1965) with anomalous-scattering correction for sulfur (Cromer, 1965); H atoms localized by difference Fourier synthesis; refined parameters: scale factor, coordinates *x*, *y*, *z*, anisotropic thermal factors of all non-hydrogen atoms; least-squares refinement based on

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