membered succinimide ring [N(1)—C(2)—C(3)—C(3′)—C(2′)]. This angle varies between 55 and 71° (Taira *et al.*, 1988; Wong & Watkins, 1973; Hambley *et al.*, 1985) compared to 44·5° for the title compound. As the relation between the structure in the crystalline state and the stable molecular conformation determined by molecular-orbital calculations has not been determined for 1(*a*) as in the case of other *N*-phenylsuccinimides, it is not clear whether this reduction is due to crystal-packing effects or molecular-electronic effects. The *N*-phenyl ring adopts a perpendicular orientation relative to the neighbouring phenyl rings in the 1,4-bis(benzylidene)-1,3-butadiene fragment (Fig. 2). The shortest distance between two neighbouring non-H atoms is C(2)···O(4)$(1-x, -y, 2-z)$ = 3·239 Å.

References

Barassin, M. J. (1963). *Ann. Chim.* (*Paris*), **8**, 637–666.

Berndt, A. F. & Schlemper, E. O. (1982). *Acta Cryst.* **B38**, 2493–2496.

Boeyens, J. C. A., Denner, L. & Perold, G. W. (1988). *J. Chem. Soc. Perkin Trans.* 2, pp. 1749–1758.

Cohen, M. D., Kaufman, H. W., Sinnreich, D. & Schmidt, G. M. J. (1970). *J. Chem. Soc. B*, pp. 1035–1039.

Crescente, O., Heller, H. G. & Oliver, S. (1979). *J. Chem. Soc. Perkin Trans.* 1, pp. 150–153.

Davidse, P. A., van Rooyen, P. H., Dillen, J. L. M., Heyns, A. M. & Modro, T. A. (1989). *Can. J. Chem.* In the press.

Hambley, T. W., Newsom, I. A. & Sternhell, S. (1985). *Aust. J. Chem.* **38**, 513–517.

Hulme, R. & Scruton, J. C. (1968). *J. Chem. Soc. A*, pp. 2448–2452.

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

Kirino, O., Takayama, C., Fujinami, A., Yanagi, K. & Minobe, M. (1984). *J. Pestic. Sci.* **9**, 351–353.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Marsh, R. E. & Donohue, J. (1967). *Adv. Protein Chem.* **22**, 235–256.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, W. S., Schonberg, A. & Singer, E. (1982). *Acta Cryst.* **B35**, 1355–1357.

Taira, Z., Takayama, C. & Terada, H. (1988). *J. Chem. Soc. Perkin Trans.* 2, pp. 1439–1445.

Van Bellingen, I., Germain, G., Piret, P. & Van Meerssche, M. (1971). *Acta Cryst.* **B27**, 560–564.

Wong, W. & Watkins, S. F. (1973). *J. Chem. Soc. Chem. Commun.* pp. 888–889.

Acta Cryst. (1990). C**46**, 1043–1046

rel-(1*R*,2*S*,3*R*,4*S*,5*S*,6*R*,7*S*,8*R*)-2,3,6,7-Tetrakis(chloromethyl)-1,4:5,8-diepoxy-1,2,3,4,5,6,7,8-octahydroanthracene

By Franz H. Kohnke,* John P. Mathias and J. Fraser Stoddart

Department of Chemistry, The University, Sheffield S3 7HF, England

and Alexandra M. Z. Slawin and David J. Williams

Department of Chemistry, Imperial College, London SW7 2AY, England

(*Received* 17 *March* 1989; *accepted* 29 *August* 1989)

Abstract. $C_{18}H_{18}Cl_4O_2$, $M_r = 408·2$, triclinic, $P\bar{1}$, $a = 6·884\,(2)$, $b = 7·782\,(2)$, $c = 9·216\,(2)$ Å, $\alpha = 101·32\,(2)$, $\beta = 93·03\,(2)$, $\gamma = 115·24\,(2)°$, $V = 433$ Å^3, $Z = 1$ (the molecule possesses a centre of symmetry), $D_x = 1·57$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1·54178$ Å, $\mu = 6·43$ mm^{-1}, $F(000) = 210$, room temperature, $R = 0·040$ for 1144 observed reflections with $|F_o| > 3\sigma(|F_o|)$. X-ray crystallography has shown that the more chromatographically mobile and higher-melting isomer – the title compound (4) – with the 2,3,6,7-tetrakis(chloromethyl)-1,4:5,8-diepoxy-1,2,3,4,5,6,7,8-octahydroanthracene constitution obtained from reaction of *cis*-1,4-dichlorobut-2-ene with the benzo[1,2-*c*:4,5-*c*′]difuran equivalent, generated *in situ* by the thermolytic decomposition of a bisadduct of 1,4:5,8-diepoxy-1,4,5,8-tetrahydroanthracene and tetraphenylcyclone, has its two O atoms oriented *anti* to each other, whilst its four chloromethyl groups all have the *endo* configuration.

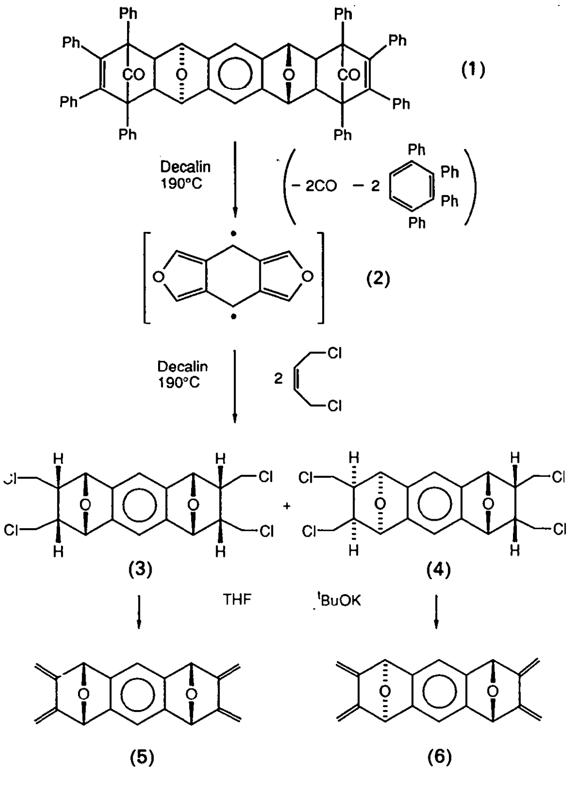
Introduction. Our current interest (Kohnke, Slawin, Stoddart & Williams, 1987; Ellwood, Mathias, Stoddart & Kohnke, 1988; Stoddart, 1988*a*,*b*, 1989;

---

* Permanent address: Dipartimento di Chimica Organica e Biologica dell' Università di Messina, Contrada Papardo, Salita Sperone, 98100 Messina, Italy.

0108-2701/90/061043-04$03.00

Kohnke & Stoddart, 1989) in the use of repetitive Diels–Alder reactions (Thomas & Miller, 1986; Chiba, Kenny & Millar, 1987) to yield polyacene and cyclacene derivatives has led us to identify *syn*-2,3,6,7-tetrakis(chloromethyl)-1,4:5,8-diepoxy-1,2,3,4,5,6,7,8-octahydroanthracene (3) and its *anti* isomer (4) as immediate precursors (see scheme) to the corresponding bisdienes (5) and (6). The tetrachlorides (3) and (4) can be obtained (Luo & Hart, 1988) as a mixture of isomers from reaction of *cis*-1,4-dichlorobut-2-ene with the benzo[1,2-*c*:4,5-*c*′]difuran equivalent (2), generated *in situ* by the thermolytic decomposition of the bisadduct (1) of 1,4:5,8-diepoxy-1,4,5,8-tetrahydroanthracene and tetraphenylcyclone. Although these isomers [(3) and (4)] can be separated (see *Experimental*) by silica gel chromatography, their relative configurations cannot be assigned unambiguously by NMR (^{1}H or ^{13}C) spectroscopies. Here, we report the full crystallographic analysis of the isomer with the higher melting point,* which is shown to have the configuration (4) with its two O atoms *anti* and its four chloromethyl groups all *endo*.



Experimental. Silica gel chromatography of an isomeric mixture of the tetrachlorides (3) and (4) using chloroform–carbon tetrachloride as eluant afforded a faster-moving component (4) and a slower-moving component (3) in approximately equal amounts. Crystals* of (3) were grown by vapour diffusion of pentane into a chloroform solution and had m.p. 465–467 K. Single crystals (found: C, 53·0; H, 4·61; Cl 34·8%; $C_{18}H_{18}Cl_4O_2$ requires C, 52·9; H, 4·44, Cl 34·8%) of (4), suitable for X-ray crystallography were grown by vapour diffusion of pentane into a dichloromethane solution and had m.p. 493 K with decomposition. Crystal size 0·17 × 0·20 × 0·30 mm. Refined unit-cell parameters obtained from setting angles of 18 reflections with $16 \leq \theta \leq 33°$. Nicolet *R*3*m* diffractometer. 1173 independent reflections ($\theta \leq 58°$) measured, Cu $K\alpha$ radiation (graphite monochromator), ω scan. 1144 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range h −7/7, k −8/8, l 0/10; two check reflections, no significant change in net counts; Lorentz and polarization corrections, numerical absorption correction (face-indexed crystal), maximum and minimum transmission factors 0·428 and 0·284. Structure solved by direct methods; non-H atoms refined anisotropically; positions of H atoms on C(1), C(2), C(3), and C(4) located from ΔF map and refined isotropically subject to a C—H distance constraint. Positions of remaining H atoms calculated (C—H = 0·96 Å), assigned isotropic thermal parameters, $U(\mathrm{H}) = 1{\cdot}2U_{\mathrm{eq}}(\mathrm{C})$, and allowed to ride on parent C atoms. An empirical extinction correction was applied [$g = 0{\cdot}023\,(5)$]. Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0{\cdot}040$; $wR = 0{\cdot}051$ [$w^{-1} = \sigma^2(F) +$

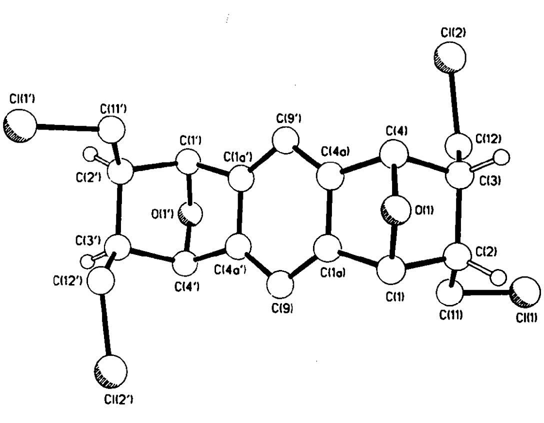


Fig. 1. Ball-and-stick representation of the structure of (4) showing the atomic numbering scheme.

---

* The isomer with the lower melting point has a severely disordered structure solvated with chloroform and could not be studied crystallographically to any degree of precision. However, it was determined unambiguously to have the *syn* configuration (3).

* Crystals of (3): $C_{18}H_{18}Cl_4O_2.xCHCl_3$, hexagonal, $P6_3$, a = 13·738 (3), c = 17·947 (4) Å, V = 2934 Å^3, Z = 6. Refinements were carried out in both $P6_3$ and $P6_3/m$, the former giving appreciably better values of R. However, severe disorder in both the x included chloroform molecules and in the chloromethyl groups prevented refinement of the structure below R = 0·13. The *syn* configuration (3) for this isomer is, however, definitive.

Table 1. *Atomic coordinates* ($\times 10^4$) *and temperature factors* ($Å^2 \times 10^3$) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
O(1)	5506 (3)	6135 (3)	1707 (2)	43 (1)
C(1)	4360 (4)	4137 (4)	1809 (3)	37 (1)
C(2)	1954 (4)	3712 (4)	1348 (3)	37 (1)
C(3)	2053 (4)	5759 (4)	2049 (3)	38 (1)
C(4)	4481 (4)	6938 (4)	2797 (3)	38 (1)
C(4*a*)	4822 (4)	6163 (3)	4112 (3)	34 (1)
C(1*a*)	4772 (4)	4358 (4)	3485 (3)	34 (1)
C(9)	4948 (4)	3142 (4)	4344 (3)	35 (1)
C(11)	382 (4)	1930 (4)	1824 (3)	41 (1)
Cl(1)	−2384 (1)	1122 (1)	972 (1)	53 (1)
C(12)	437 (4)	5761 (4)	3088 (3)	44 (1)
Cl(2)	960 (1)	8209 (1)	4012 (1)	63 (1)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(1)	1·439 (3)	O(1)—C(4)	1·450 (4)
C(1)—C(2)	1·561 (4)	C(1)—C(1*a*)	1·517 (3)
C(2)—C(3)	1·568 (4)	C(2)—C(11)	1·515 (4)
C(3)—C(4)	1·559 (3)	C(3)—C(12)	1·507 (4)
C(4)—C(4*a*)	1·505 (4)	C(4*a*)—C(1*a*)	1·395 (4)
C(4*a*)—C(9*a*)	1·394 (3)	C(1*a*)—C(9)	1·385 (4)
C(9)—C(4*a*′)*	1·394 (3)	C(11)—Cl(1)	1·807 (3)
C(12)—Cl(2)	1·797 (3)		
C(1)—O(1)—C(4)	96·5 (2)	O(1)—C(1)—C(2)	101·2 (2)
O(1)—C(1)—C(1*a*)	101·4 (2)	C(2)—C(1)—C(1*a*)	108·9 (2)
C(1)—C(2)—C(3)	100·7 (2)	C(1)—C(2)—C(11)	111·3 (3)
C(3)—C(2)—C(11)	118·9 (2)	C(2)—C(3)—C(4)	101·0 (2)
C(2)—C(3)—C(12)	116·0 (2)	C(4)—C(3)—C(12)	114·7 (2)
O(1)—C(4)—C(3)	100·5 (2)	O(1)—C(4)—C(4*a*)	101·5 (2)
C(3)—C(4)—C(4*a*)	108·9 (2)	C(4)—C(4*a*)—C(1*a*)	105·1 (2)
C(4)—C(4*a*)—C(9*a*)	133·1 (3)	C(1*a*)—C(4*a*)—C(9*a*)	121·8 (3)
C(1)—C(1*a*)—C(4*a*)	104·1 (2)	C(1)—C(1*a*)—C(9)	133·2 (2)
C(4*a*)—C(1*a*)—C(9)	122·7 (2)	C(1*a*)—C(9)—C(4*a*′)*	115·6 (2)
C(2)—C(11)—Cl(1)	111·7 (2)	C(3)—C(12)—Cl(2)	111·6 (2)

*These atoms are related to those in Table 1 by the operation $1-x$, $1-y$, $1-z$.

$0{\cdot}00029F^2$]; $S = 3{\cdot}13$; $(\Delta/\sigma)_{\max} = 0{\cdot}018$; residual electron density in difference synthesis within $-0{\cdot}26$ and $0{\cdot}42$ e Å^{-3}; atomic scattering factors and dispersion corrections from *International Tables for X-ray Crystallography* (1974). Computations carried out on an Eclipse S140 computer using the *SHELXTL* program system (Sheldrick, 1983).

Discussion. The structure of (4) with atomic numbering scheme is shown in Fig. 1. Table 1* lists the fractional atomic coordinates and the non-H atoms and Table 2 the bond lengths and angles.

The tetrachloride (4) possesses crystallographic C_i symmetry with the four chloromethyl substituents having the *endo* configuration, which is in common with that observed in the poorly refined *syn* tetrachloride (3). Thus, the reaction (see scheme) of *cis*-1,4-dichlorobut-2-ene with the benzo[1,2-*c*:4,5-*c*′]-difuran equivalent (2) is highly diastereoselective with respect to each furan ring in (2). However, the dienophiles approach the diene units in (2) with more

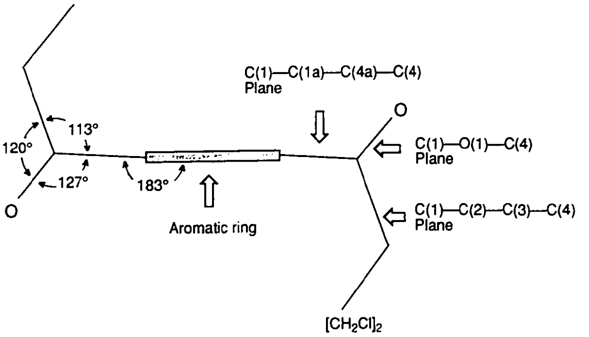


Fig. 2. Diagrammatic representation of the structure of (4) defining space around the molecule, mean planes in the molecule, and showing dihedral angles.

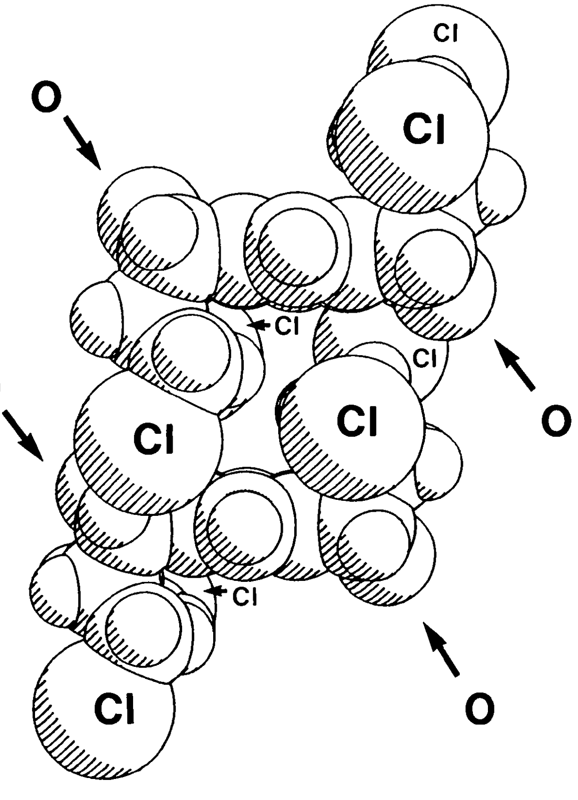


Fig. 3. Space-filling representation of the packing of a centrosymmetrically related pair of molecules.

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52549 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

or less a similar probability from the same and opposite faces of (2), leading to the formation of (3) and (4), respectively, in approximately equal quantities. The absence of C_{2h} symmetry and the essentially orthogonal relationship of the C—Cl bonds in the vicinal C(2)—C(3) positions is probably a consequence of otherwise unfavourable steric interactions. The C(2)—C(3)—C(12)—Cl(2) and the C(3)—C(2)—C(11)—Cl(1) torsion angles are 172·0 (2) and 75·6 (2)°, respectively. The dihedral angles (Fig. 2) between C(1)—O(1)—C(4) and the C(1)—C(1*a*)—C(4*a*)—C(4) and the C(1)—C(2)—C(3)—C(4) mean planes are 127 (1) and 120 (1)°, respectively and that between the C(1)—C(2)—C(3)—C(4) and the C(1)—C(1*a*)—C(4*a*)—C(4) mean planes is 113 (1)°. The C(1)—O(1)—C(4) plane is canted further away from the mean plane of the aromatic ring by an extra 3°. These molecular deformations are virtually identical to those observed (Kohnke, Stoddart, Slawin & Williams, 1988) in methyl *rel*-(1*R*,2*R*,3*S*,4*S*,5*S*,6*S*,7*R*,8*R*)-1,4:5,8-diepoxy-1,2,3,4,5,6,7,8-octahydroanthracene-2,3,6,7-tetracarboxylate.

The packing is normal van der Waals with no short intermolecular contacts, the molecules interleaving with each other in the crystallographic **a** direction (Fig. 3).

We thank the Ministry of Defence and the Science and Engineering, and Agricultural and Food Research Councils in the United Kingdom, and the University of Messina in Italy, for financial support.

References

Chiba, T., Kenny, P. W. & Miller, L. L. (1987). *J. Org. Chem.* **52**, 4327–4331.
Ellwood, P., Mathias, J. P., Stoddart, J. F. & Kohnke, F. H. (1988). *Bull. Soc. Chim. Belg.* **97**, 669–678.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Kohnke, F. H., Slawin, A. M. Z., Stoddart, J. F. & Williams, D. J. (1987). *Angew. Chem. Int. Ed. Engl.* **26**, 892–894.
Kohnke, F. H. & Stoddart, J. F. (1989). *Pure Appl. Chem.* **61**, 1581–1586.
Kohnke, F. H., Stoddart, J. F., Slawin, A. M. Z. & Williams, D. J. (1988). *Acta Cryst.* **C44**, 740–742.
Luo, J. & Hart, H. (1988). *J. Org. Chem.* **53**, 1341–1343.
Sheldrick, G. M. (1983). *SHELXTL*, revision 4.0, January 1983. *An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.
Stoddart, J. F. (1988*a*). *Nature* (*London*), **334**, 10–11.
Stoddart, J. F. (1988*b*). *Chem. Br.* **24**, 1203–1208.
Stoddart, J. F. (1989). *J. Inclusion Phenom.* **7**, 227–245.
Thomas, A. D. & Miller, L. L. (1986). *J. Org. Chem.* **51**, 4160–4169.

Acta Cryst. (1990). **C46**, 1046–1049

rel-(1*R*,4*S*,5*R*,8*S*)-1,4:5,8-Diepoxy-1,4,5,8-tetrahydro-2,3,6,7-tetramethyleneanthracene

By Franz H. Kohnke,* John P. Mathias and J. Fraser Stoddart
Department of Chemistry, The University, Sheffield S3 7HF, England

and Alexandra M. Z. Slawin, David J. Watts and David J. Williams
Department of Chemistry, Imperial College, London SW7 2AY, England

(*Received* 17 *March* 1989; *accepted* 25 *September* 1989)

Abstract. $C_{18}H_{14}O_2$, $M_r = 262{\cdot}3$, monoclinic, $P2_1/n$, $a = 10{\cdot}567\ (2)$, $b = 8{\cdot}149\ (2)$, $c = 16{\cdot}128\ (3)$ Å, $\beta = 104{\cdot}33\ (2)°$, $V = 1346$ Å^3, $Z = 4$, $D_x = 1{\cdot}29$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}54178$ Å, $\mu = 0{\cdot}63$ mm^{-1}, $F(000) = 552$, room temperature, $R = 0{\cdot}053$ for 1632 observed reflections with $|F_o| > 3\sigma(|F_o|)$. Symmetry-related pairs of molecules of the title compound (5), prepared stereospecifically from the *syn* tetrachloride, pack so that an olefinic H atom in one molecule interacts electrostatically with the π system on the *endo* face of another molecule.

Introduction. The bisdiene (5) with the *syn* configuration is a key compound in the synthesis of polyacene and cyclacene derivatives using repetitive Diels–Alder reactions (Thomas & Miller, 1986; Chiba, Kenny & Miller, 1987). We have now obtained this compound as a pure diastereoisomer (5) stereospecifically from treatment of the *syn* tetrachloride (3) (Kohnke, Mathias, Stoddart, Slawin & Williams,

---

* Permanent address: Dipartimento di Chimica Organica e Biologica dell' Università di Messina, Contrada Papardo, Salita Sperone, 98100 Messina, Italy.

0108-2701/90/061046-04$03.00