122·7 (6); 119·1 (6); 123·4 (6)°]. *tert*-Butoxycarbonyl-
L-alanyl-L-asparaginyl-L-proline benzyl ester
[120·7 (3)°], and Boc-Asn^1-Pro-^{2}-Ser^3(Bzl)-NHMe
and Boc-Asn^1(Me)-Pro^2-Ser^3(Bzl)-NHMe [120·5 (4)°;
121·5 (4)°]. Torsion angles C7—C8—C9—C10
[34·7 (2)°], C12—C13—C14—C15 [38·1 (3)°], and
O1—C1—C2—O2 [− 34·9 (2)°] of the title molecule
show the similarity of the ring conformation adopted
by the heterocyclic rings; namely, the two pyrrolidine
and the 1,3-dioxolane rings.

References

AUBRY, A., ABBADI, A., BOUSSARD, G. & MARRAUD, M. (1987).
    Nouv. J. Chim. **11**, 739–744.

CROMER, D. T. (1974). *International Tables for X-ray Crystallogra-
    phy*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present
    distributor Kluwer Academic Publishers, Dordrecht.)
CROMER, D. T. & WABER, J. T. (1974). *International Tables for
    X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham:
    Kynoch Press. (Present distributor Kluwer Academic
    Publishers, Dordrecht.)
DOCK, A.-C., MORAS, D., BEHR, J.-P. & LEHN, J. M. (1983). *Acta
    Cryst.* C39, 1001–1005.
FRENZ, B. A. & OKAYA, Y. (1980). *Enraf–Nonius Structure Deter-
    mination Package*. Enraf–Nonius, Delft, The Netherlands.
IN, Y., DOI, M., ISHIDA, T., INOUE, M. & TOMITA, K.-I. (1987).
    Acta Cryst. C43, 770–773.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge
    National Laboratory, Tennessee, USA.
KLEIN, U., MOHRS, K., WILD, H. & STEGLICH, W. (1987). *Ann.
    Chem.* pp. 485–489.
MUSICH, J. A. & RAPOPORT, H. (1978). *J. Am. Chem. Soc.* **100**,
    4865–4872.
SEEBACH, D., KALINOWSKI, H. O., BASTANI, B., CRASS, G., DAUM,
    H., DÖRR, H., DUPREEZ, N. P., EHRIG, V., LANGER, W.,
    NÜSSLER, C., OEI, H.-A. & SCHMIDT, M. (1977). *Helv. Chim.
    Acta*, **60**, 301–325.

trans-1,5-Bicyclo[4.4.0]decadione*

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Abstract. $C_{10}H_{14}O_2$, $M_r = 166·2$, monoclinic, $P2_1/n$, $a = 7·4465$ (6), $b = 5·5949$ (3), $c = 10·9675$ (14) Å, $\beta = 106·776$ (8)°, $V = 437·48$ (7) Å^3, $Z = 2$, $D_x = 1·262$ g cm^{-3}; λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 6·6$ cm^{-1}, $F(000) = 180$, $T = 296$ K, $R = 0·033$ for 744 observa-
tions with $I > 3\sigma(I)$ (of 899 unique data). The mol-
ecule lies on an inversion center, and the *trans* fused
cyclohexanone rings are in the chair conformation.
The C=O bond length is 1·213 (2) Å, and the central
bond has length 1·5474 (13) Å.

Experimental. The title compound was prepared by
treating decalin-1,5-diol with 8N chromic acid in
acetone at room temperature (Peet & Cargill, 1973).
The *trans* isomer was formed by treatment with
hydrochloric acid in chloroform (Hamon & Rich-
ards, 1973; Kleinfelter & Schleyer, 1973).

Crystals that deposited from the reaction flask,
m.p. 426–428 K, were suitable; a clear colorless crys-
tal with dimensions 0·12 × 0·20 × 0·30 mm was used
for data collection on an Enraf–Nonius CAD-4 dif-
fractometer with Cu $K\alpha$ radiation and a graphite
monochromator. Cell dimensions were determined
from setting angles of 25 reflections having $30 > \theta >
25°$. The ω–2θ scans were designed for $I = 50\sigma(I)$,
subject to maximum scan time = 120 s, scan rates
varied in the range 0·57–3·30° min^{-1}. A full sphere
of data having $2 < \theta < 75°$, $-9 \leq h \leq 9$, $-7 \leq k \leq 7$,
$-13 \leq l \leq 13$ was measured and corrected for back-
ground, Lorentz and polarization effects, decay, and
absorption. Absorption corrections were based on ψ
scans, with minimum relative transmission coefficient
97·2%. Three standard reflections (200, 040, 006)
decreased in intensity by 15·7% due to sublimation,
and a linear correction was applied. A total of 3452
data was measured. Data from the four equivalent
quadrants were averaged, $R_{int} = 0·013$, yielding 899
unique data. Systematic absences 0k0 with k odd and
h0l with $h + l$ odd indicated space group $P2_1/n$. The
structure was solved by direct methods, refined by
full-matrix least squares based upon F, using data for
which $I > 3\sigma)I)$, weights $w = 4F_o^2[\sigma^2(I) +
(0·02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Deter-
mination Package* (Frenz & Okaya, 1980), scattering
factors of Cromer & Waber (1974), and anomalous
coefficients of Cromer (1974). C-atom coordinates
were refined with anisotropic thermal parameters;

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* *trans*-Decalin-1,5-dione.
† Author to whom correspondence should be addressed.

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
O	0·0872 (1)	0·0944 (2)	0·67140 (9)	5·73 (2)
C1	0·1327 (2)	0·2525 (2)	0·6106 (1)	3·71 (2)
C2	0·0146 (1)	0·4753)2)	0·57162 (8)	3·07 (2)
C3	−0·1714 (2)	0·4670 (3)	0·6037 (1)	4·09 (2)
C4	−0·2782 (2)	0·7000 (3)	0·5679 (1)	4·48 (3)
C5	0·3101 (2)	0·2397 (2)	0·5725 (1)	4·47 (3)

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

O—C1	1·213 (2)	C2—C2'	1·5474 (13)
C1—C2	1·514 (2)	C2—C3	1·525 (2)
C1—C5	1·498 (2)	C3—C4	1·518 (2)
C4—C5'	1·526 (2)		

O—C1—C2	121·57 (12)	C2'—C2—C3	111·63 (7)
O—C1—C5	121·82 (11)	C2—C3—C4	110·92 (11)
C2—C1—C5	116·60 (10)	C3—C4—C5'	111·60 (11)
C1—C2—C2'	109·49 (9)	C1—C5—C4	112·19 (9)
C1—C2—C3	113·71 (10)		

O—C1—C2—C3	−6·0 (2)	O—C1—C2—C2'	−131·7 (1)
C5—C1—C2—C3	175·2 (1)	C5—C1—C2—C2'	49·5 (1)
C2—C1—C5—C4'	−48·9 (1)	C1—C2—C3—C4	177·3 (1)
C2'—C2—C3—C4	−58·2 (1)	C1—C2—C2'—C3'	−53·2 (1)
C2—C3—C4—C5'	56·2 (1)	C1—C5—C4'—C3'	50·6 (2)

H-atom coordinates were located by ΔF and were refined with isotropic thermal parameters. Final $R = 0·033$ for 744 observed data (0·040 for all 899 data), $wR = 0·049$, and $S = 2·533$ for 84 variables. Maximum shift $< 0·01\sigma$ in the final cycle, max. residual density 0·13, min. $-0·09$ e Å^{-3}, and extinction coefficient $g = 2·2\ (2) \times 10^{-5}$ where the factor $(1 + gI_c)^{-1}$ was applied to F_c. A structural diagram of the title compound is shown below while the fractional coordinates are given in Table 1. Fig. 1 is a perspective drawing showing the atom numbering. Fig. 2 illustrates the unit cell. Bond distances, angles, and selected torsion angles are presented in Table 2.*
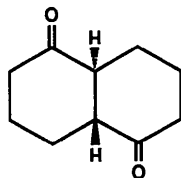
Related literature. The structure of *trans*-decalin-1,4-dione (Ariel & Trotter, 1984) shows similar bond lengths and bond angles.

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* Lists of H-atom coordinates and thermal parameters, bond distances and angles involving H, structure-factor amplitudes, anisotropic thermal parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53296 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
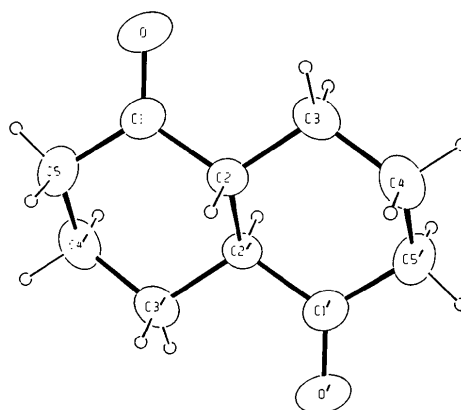

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius.
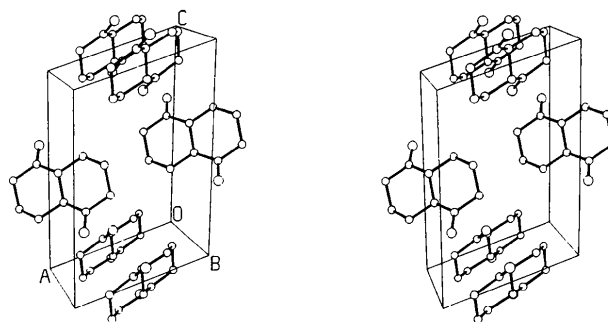

Fig. 2. Stereoview of the unit cell.

The cyclohexanone rings have a chair conformation, with torsion angles ranging from ±48·9 (1) to ±58·2 (1)°, that is slightly distorted from the ideal chair conformation having torsion angles of ±55° and valence angles of 111·5° (Bucourt & Hainaut, 1965).

References

ARIEL, S. & TROTTER, J. (1984). *Acta Cryst.* C40, 1253–1255.
BUCOURT, R. & HAINAUT, D. (1965). *Bull. Soc. Chim. Fr.* pp. 1366–1378.
CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
FRENZ, B. A. & OKAYA, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

HAMON, D. P. G. & RICHARDS, K. R. (1973). *Aust. J. Chem.* **36**, 2243–2259.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

KLEINFELTER, D. C. & SCHLEYER, P. VON R. (1973). *Org. Synth.* **5**, 852–855.

PEET, N. P. & CARGILL, R. L. (1973). *J. Org. Chem.* **38**, 4281–4285.

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endo–endo Diels–Alder Diadduct of 5,5-Dimethoxy-1,2,3,4-tetrachloro-1,3-cyclopentadiene with 1,5-Cyclooctadiene, (1), and the Dechlorinated Diels–Alder Diadduct, (2)

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Abstract. 1,6,7,8,9,14,15,16-Octachloro-17,17,18,18-tetramethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (1), $C_{22}H_{24}Cl_8O_4$, $M_r = 636.1$, triclinic, $P\bar{1}$, $a = 7.3110$ (7), $b = 7.9195$ (5), $c = 12.2113$ (10) Å, $\alpha = 84.490$ (7), $\beta = 86.064$ (7), $\gamma = 71.490$ (8)°, $V = 666.8$ (1) Å^3, $Z = 1$, $D_x = 1.584$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1.54184$ Å, $\mu = 81.7$ cm^{-1}, $F(000) = 324$, $T = 296$ K, $R = 0.043$ for 2259 observations with $I > 3\sigma(I)$ (of 2659 unique data). 17,17,18,18-Tetramethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (2), $C_{22}H_{32}O_4$, $M_r = 360.5$, triclinic, $P\bar{1}$, $a = 6.5121$ (7), $b = 8.5391$ (8), $c = 9.4741$ (4) Å, $\alpha = 67.293$ (6), $\beta = 76.580$ (6), $\gamma = 88.202$ (8)°, $V = 471.7$ (1) Å^3, $Z = 1$, $D_x = 1.269$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1.54184$ Å, $\mu = 6.45$ cm^{-1}, $F(000) = 196$, $T = 296$ K, $R = 0.036$ for 1815 observations with $I > 3\sigma(I)$ (of 1942 unique data). The *endo–endo* isomer (1) of the four possible Diels–Alder diadducts is preferred at lower temperature. Both molecules lie on inversion centers, thus the cyclooctane ring in both structures adopts the chair conformation. The near-zero torsion angles of the chair occur at the ring-fusion bonds, and have magnitudes of 1.8 (4)° for the chlorinated compound (1) and 0.89 (13)° for the dechlorinated compound (2). The C=C bond has a length of 1.324 (3) in (1) and 1.329 (1) Å in (2).

Experimental. The title compound (1) was prepared by heating a 4:1 mixture of 5,5-dimethoxy-1,2,3,4-tetrachloro-1,3-cyclopentadiene with 1,5-cyclooctadiene to 453 K and extracting the white solid with pyridine (Akhtar, Fray & Yarrow, 1968). Crystals of the *endo–endo* isomer (1), grown by slow cooling of xylenes, m.p. 629–631 K (dec.), were suit-,

able; a clear colorless crystal with dimensions 0.10 × 0.20 × 0.22 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $30 > \theta > 11°$. The ω–2θ scans were designed for $I = 25\sigma(I)$, subject to maximum scan time = 120 s, scan rates varied in the range 0.72–3.30° min^{-1}. One hemisphere of data having $2 < \theta < 75°$, $0 \le h \le 9$, $-9 \le k \le 9$, $-15 \le l \le 15$ was measured and corrected for background, Lorentz and polarization effects, decay, and absorption. Absorption corrections were based on ψ scans, with minimum relative transmission coefficient 0.574. Three standard reflections (300, 020, 003) were measured every 10 000 s, exhibiting 19, 53, and 20% intensity decline, respectively. An anisotropic decay correction was applied. The structure was solved by direct methods, refined by full-matrix least squares based upon F, using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). C-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF and were refined with isotropic thermal parameters. Final $R = 0.043$ for 2259 observed data (0.059 for all 2659 data), $wR = 0.059$, and $S = 3.303$ for 203 variables. Maximum shift 0.15σ in the final cycle, max. residual density 0.30, min. -0.48 e Å^{-3}, and extinction coefficient $g = 8.4$ (6) × 10^{-6} where the factor $(1 + gI_c)^{-1}$ was applied to F_c. A structural diagram of compound (1) is shown below while the fractional coordinates are given in Table 1. Fig. 1 is a perspective drawing showing the atom numbering for title molecule (1),

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