SLETTEN, J. & JENSEN, L. H. (1969). *Acta Cryst.* B25, 1608–1614.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.

VOET, D. & RICH, A. (1970). *Prog. Nucleic Acid Res. Mol. Biol.* 10, 183–265.

WEHE, D. J., BUSING, W. R. & LEVY, H. A. (1962). *ORABS.* Report ORNL-TM-229. Oak Ridge National Laboratory, Tennessee.

WILSON, A. J. C. (1942). *Nature (London)*, 150, 151–152.

ZALKIN, A. (1965). *FORDAP.* Univ. of California, Berkeley, USA.

1,3,4,5-*endo*,7,7-Hexachloro-2-azabicyclo[2.2.1]hept-2-ene: A Diels–Alder Adduct of Vinyl Chloride and 2,3,4,5,5-Pentachloro-1-azacyclopentadiene

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Abstract. $C_6H_3Cl_6N$, monoclinic, $P2_1/n$, $Z = 4$, $a = 6.179 (4)$, $b = 14.799 (6)$, $c = 11.693 (4)$ Å, $\beta = 92.25 (5)°$, $D_c = 1.876$ Mg m^{-3}. The structure was solved by direct phasing methods and refined to $R = 0.051$ for 1961 independent reflections. Both bridge-heads are C—Cl. The bicyclo ring structure contains a —N=CCl moiety.

Introduction. The title compound was prepared by Gladstone, Daniels & Wong (1977) as part of a program investigating Diels–Alder reactions involving 1-azadienes.
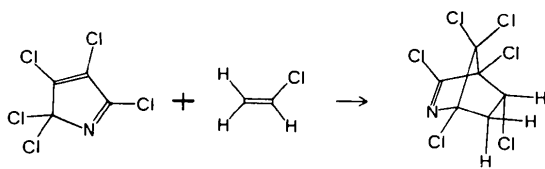
Without the heterocyclic N present, similar reactions are used to prepare the commercial insecticides aldrin and dieldrin. These reactions are both stereo- and regio-specific, yielding only the *endo*-5-substituted azanor-bornenes. This work shows that in adduct (4) of the above reference (which is the title compound) the N is located at position 2, rather than the originally assigned position 1.

Translucent crystals with good face development were obtained from Gladstone, Daniels & Wong (1977). X-ray diffraction data showed the crystals to be monoclinic, with systematic absences $h0l$: $h + l = 2n$ and $0k0$: $k = 2n$, space group $P2_1/n$. The alternative space group assignment of $P2_1/c$ leads to a very large β angle.

1961 independent reflections were collected with an Enraf–Nonius CAD-4 diffractometer (Mo $K\alpha$ radiation). The crystal exhibited darkening as a result of X-ray exposure; however, three reference reflection intensities remained essentially constant through the data collection. Lorentz and polarization corrections were made in the usual manner and the data were reduced to observed structure factor amplitudes.

The phase problem was solved by symbolic addition with the program *SHELX* 76 (Sheldrick, 1976). 232 reflections with $E > 1.1$ were used with a starting set of 17 reflections. An analysis for sign indeterminance in the best four solutions led to the omission of 10 reflections from the starting set. After the selection of a new starting set, two solutions were calculated, one containing a recognizable molecular fragment. A structure factor calculation on the 10 highest peaks ($R = 41\%$), followed by a difference Fourier map, was sufficient to locate the remaining three heavy atoms.

The structure was refined by the full-matrix least-squares program *ORXFLS*3 (Busing, Martin & Levy, 1971) with isotropic and then anisotropic temperature factors, until $R = 0.067$. H atoms were found on a difference Fourier map and refined with isotropic temperature factors. H scattering factors were those of Stewart, Davidson & Simpson (1965). A final conventional R factor of 0.051 ($R_w = 0.082$) was obtained, excluding unobserved reflections (215 reflections with $F_0 < 3\sigma$). The final difference Fourier map displayed no distinguishing features.*

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34420 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final positional parameters* ($\times 10^4$, *for* H $\times 10^3$) *with standard deviations*

	x	y	z
Cl(1)	3456 (2)	5567 (1)	2001 (1)
Cl(3)	4014 (2)	2521 (1)	4315 (1)
Cl(4)	8366 (2)	2493 (1)	2736 (1)
Cl(5)	8536 (2)	3892 (1)	5138 (1)
Cl(7)	8258 (2)	4512 (1)	1136 (1)
Cl(7')	4408 (2)	3441 (1)	977 (1)
C(1)	5008 (5)	4723 (2)	2694 (3)
N(2)	3662 (4)	4130 (2)	3390 (2)
C(3)	4788 (5)	3425 (2)	3528 (3)
C(4)	6952 (5)	3489 (2)	2977 (3)
C(5)	8243 (5)	4214 (2)	3672 (3)
C(6)	6865 (5)	5069 (2)	3477 (3)
C(7)	6165 (5)	4043 (2)	1924 (3)
H(5)	981 (8)	428 (3)	344 (4)
H(6)	776 (8)	556 (3)	310 (4)
H(6')	625 (8)	525 (3)	411 (4)
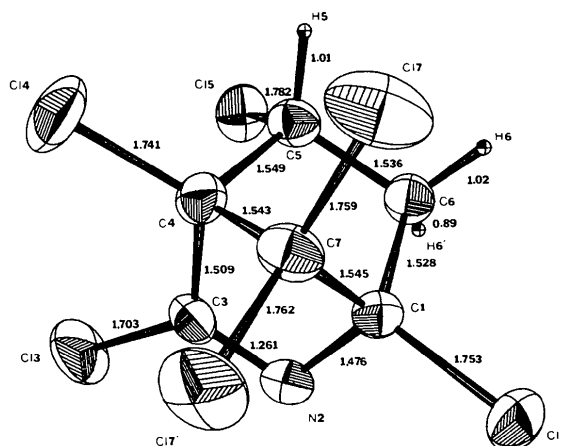
Fig. 1. The molecular structure, showing the atomic numbering system and the bond lengths (Å). The thermal ellipsoids enclose 50% probability (Johnson, 1965).

Table 2. *Final bond angles* (°) *with standard deviations*

N(2)C(1)C(6)	107·2 (3)	N(2)C(1)C(7)	102·6 (3)
N(2)C(1)Cl(1)	111·6 (2)	C(6)C(1)C(7)	102·3 (3)
C(6)C(1)Cl(1)	115·0 (2)	C(7)C(1)Cl(1)	116·9 (2)
C(1)N(2)C(3)	104·0 (3)	N(2)C(3)C(4)	112·8 (3)
N(2)C(3)Cl(3)	123·6 (3)	C(4)C(3)Cl(3)	123·5 (2)
C(3)C(4)Cl(4)	118·3 (2)	C(3)C(4)C(7)	96·7 (3)
C(3)C(4)C(5)	105·5 (2)	C(7)C(4)Cl(4)	117·7 (2)
C(5)C(4)Cl(4)	114·8 (2)	C(7)C(4)C(5)	100·9 (2)
C(4)C(5)C(6)	102·8 (3)	C(4)C(5)Cl(5)	110·5 (2)
C(6)C(5)Cl(5)	113·5 (2)	C(4)C(5)H(5)	113 (2)
Cl(5)C(5)H(5)	103 (3)	C(6)C(5)H(5)	114 (2)
C(1)C(6)C(5)	102·2 (3)	C(5)C(6)H(6)	110 (3)
C(5)C(6)H(6')	112 (3)	C(1)C(6)H(6)	112 (3)
C(1)C(6)H(6')	106 (3)	H(6)C(6)H(6')	114 (4)
C(1)C(7)C(4)	91·1 (2)	C(1)C(7)Cl(7)	114·5 (2)
C(1)C(7)Cl(7')	114·1 (2)	C(4)C(7)Cl(7)	114·4 (2)
C(4)C(7)Cl(7')	113·5 (2)	Cl(7)C(7)Cl(7')	108·6 (2)

Discussion. The final atomic coordinates are given in Table 1. A thermal-ellipsoid plot (Johnson, 1965) of the molecule, with the atomic numbering system, is shown in Fig. 1. The bond distances are also shown in the figure; the estimated standard deviations are 0·003, 0·004, 0·004 and 0·045 Å for C—Cl, C—C, C—N and C—H respectively. The bond angles are given in Table 2.

The average C—Cl bond distance for the five Cl atoms bonded to sp^3 C atoms is 1·759 Å. As expected, the C—Cl bond distance to the sp^2 C atom is shorter at 1·703 Å. The N(2)—C(3) bond length of 1·261 Å clearly indicates the presence of a double bond.

Two bond angles deviate more than 10° from either tetrahedral or trigonal values. The CCl₂ bridging group forms a C(1)C(7)C(4) angle of 91·1°, because of ring strain. Also, the C(3)C(4)C(7) angle of 96·7° shows the presence of bridgehead strain, caused by the double bond in the bicyclo system. C(1), N(2), C(3), Cl(3) and C(4) are coplanar within 0·021 Å.

The shortest intermolecular distance is a N···H at 2·39 Å. The shortest Cl···Cl intermolecular distance is 3·47 Å.

References

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1971). *ORXFLS*3. Oak Ridge National Laboratory, Tennessee.

GLADSTONE, C. M., DANIELS, P. H. & WONG, J. L. (1977). *J. Org. Chem.* **42**, 1375–1379.

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

SHELDRICK, G. M. (1976). *SHELX* 76. A program for crystal structure determination. Univ. of Cambridge, England.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.