Table 2. *Interatomic distances* (Å) *and angles* (°)

(*a*) Bond lengths

C(1)–C(2)	1·39 (1)	C(9)–C(17)	1·48 (1)
C(1)–C(6)	1·41 (1)	C(10)–C(11)	1·52 (1)
C(1)–C(7)	1·43 (1)	C(10)–N(3)	1·36 (1)
C(2)–C(3)	1·35 (2)	C(11)–N(1)	1·46 (1)
C(3)–C(4)	1·40 (2)	C(11)–N(2)	1·46 (1)
C(4)–C(5)	1·40 (1)	C(13)–N(2)	1·47 (1)
C(5)–C(6)	1·35 (1)	C(14)–N(2)	1·49 (1)
C(6)–N(1)	1·39 (1)	C(15)–N(3)	1·47 (1)
C(7)–C(8)	1·37 (1)	C(16)–N(3)	1·47 (1)
C(7)–C(12)	1·51 (1)	C(17)–O(1)	1·35 (1)
C(8)–C(9)	1·46 (1)	C(17)–O(2)	1·21 (1)
C(8)–N(1)	1·40 (1)	C(18)–O(1)	1·43 (1)
C(9)–C(10)	1·37 (1)		

(*b*) Bond angles

C(2)–C(1)–C(6)	117·5 (10)	C(9)–C(10)–C(11)	111·0 (7)
C(2)–C(1)–C(7)	133·6 (10)	C(9)–C(10)–N(3)	129·5 (9)
C(6)–C(1)–C(7)	108·8 (7)	C(11)–C(10)–N(3)	119·4 (9)
C(1)–C(2)–C(3)	119·6 (11)	C(10)–C(11)–N(1)	101·1 (7)
C(2)–C(3)–C(4)	122·1 (11)	N(1)–C(11)–N(2)	116·1 (7)
C(3)–C(4)–C(5)	119·5 (11)	C(10)–C(11)–N(2)	110·0 (8)
C(4)–C(5)–C(6)	117·3 (11)	C(9)–C(17)–O(1)	110·9 (9)
C(1)–C(6)–C(5)	124·1 (9)	C(9)–C(17)–O(2)	126·1 (10)
C(1)–C(6)–N(1)	106·0 (8)	O(1)–C(17)–O(2)	122·9 (10)
C(5)–C(6)–N(1)	129·9 (10)	C(6)–N(1)–C(8)	109·4 (7)
C(1)–C(7)–C(8)	106·8 (8)	C(6)–N(1)–C(11)	136·5 (8)
C(1)–C(7)–C(12)	123·5 (8)	C(8)–N(1)–C(11)	112·4 (7)
C(8)–C(7)–C(12)	129·7 (10)	C(11)–N(2)–C(13)	114·0 (9)
C(7)–C(8)–N(1)	108·9 (8)	C(11)–N(2)–C(14)	112·4 (8)
C(9)–C(8)–N(1)	107·2 (8)	C(13)–N(2)–C(14)	113·5 (10)
C(7)–C(8)–C(9)	142·8 (10)	C(10)–N(3)–C(15)	123·2 (9)
C(8)–C(9)–C(10)	108·2 (8)	C(10)–N(3)–C(16)	123·4 (9)
C(8)–C(9)–C(17)	121·6 (9)	C(15)–N(3)–C(16)	113·4 (8)
C(10)–C(9)–C(17)	129·4 (9)	C(17)–O(1)–C(18)	117·2 (9)

(*c*) Torsion angles

C(1)–C(2)–C(3)–C(4)	0·1	C(7)–C(1)–C(6)–N(1)	2·2
C(2)–C(3)–C(4)–C(5)	1·3	N(1)–C(8)–C(9)–C(10)	1·5
C(3)–C(4)–C(5)–C(6)	2·0	C(8)–C(9)–C(10)–C(11)	−2·1
C(4)–C(5)–C(6)–C(1)	1·7	C(9)–C(10)–C(11)–N(1)	1·8
C(5)–C(6)–C(1)–C(2)	0·6	C(10)–C(11)–N(1)–C(8)	0·8
C(6)–C(1)–C(2)–C(3)	0·3	C(11)–N(1)–C(8)–C(9)	−0·3
C(1)–C(6)–N(1)–C(8)	−3·0	C(2)–C(1)–C(6)–N(1)	179·3
C(6)–N(1)–C(8)–C(7)	2·8	C(7)–C(1)–C(6)–C(5)	−176·5
N(1)–C(8)–C(7)–C(1)	−1·3	C(7)–C(8)–N(1)–C(11)	170·4
C(8)–C(7)–C(1)–C(6)	0·5	C(6)–N(1)–C(8)–C(9)	−168·0

The standard deviations in torsion angles are in the range 1·2 to 1·6° for torsion angles near zero and 1·8 to 2·6° for those angles near 180°.

Discussion. The molecule shows signs of intramolecular overcrowding. The benzene ring, C(1)–C(6), is close to being planar, but the five-membered ring N(1),C(8)–C(11) adopts a slight envelope conformation with C(9) at the flap, to reduce crowding between the CO_2Me moiety on C(9) and the methyl atoms C(12) and C(16) [*cf.* C(11)–C(10)–N(3) 119·4 (9)°, C(9)–C(10)–N(3) 129·5 (9)° and C(1)–C(7)–C(12) 123·5 (8)° and C(8)–C(7)–C(12) 129·7 (10)°]. As a consequence, the molecule is slightly folded and there is a 12·5° angle between the benzene and N(1),C(8)–C(11) ring planes.

Molecular dimensions are normal within the accuracy of the analysis (*e.g.* mean aromatic C–C 1·40 Å, mean N–Me 1·50 Å). The bond lengths C(7)–C(8) 1·37 (1), C(8)–C(9) 1·46 (1), C(9)–C(10) 1·37 (1) Å, together with the orange colour of (2), and the UV spectrum (Jones *et al.*, 1974), suggest some delocalization involving the C(7), C(8) and C(9), C(10) double bonds with the benzenoid ring system. In the crystal structure, molecules are separated by normal van der Waals distances.

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5-*endo*-(Bromomethyl)-1,2,4-trichloro-3,7,7-trimethoxynorborn-2-ene*

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Abstract. $C_{11}H_{14}BrCl_3O_3$, monoclinic, $P2_1/n$, $Z = 4$, $a = 7·988$ (3), $b = 15·672$ (8), $c = 11·949$ (4) Å, $\beta = 103·90$ (3)°, $D_c = 1·74$ Mg m^{-3}; $R_w = 0·071$ for 1478 observed reflections. The geometry of the title compound is compared with a number of closely related molecules. Anomeric interaction governs the shape of the CH_3–O–C(7)–O–CH_3 moiety. The bicyclo-[2.2.1]hept-2-ene skeleton is hardly distorted, despite heavy substitution. Rather the geometry of substituents is influenced by the skeleton causing the inequivalence of many seemingly equivalent valence

* Norbornane has been superseded by the name 8,9,10-trinorbornane.

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angles, *e.g.* C(4)—C(7)—O(8) and C(1)—C(7)—O(9) are about 10° larger than C(4)—C(7)—O(9) and C(1)—C(7)—O(8).

Introduction. In continuation of research on heavily substituted norborn-2-ene systems we undertook the structure determination of 5-(bromomethyl)-1,2,4-trichloro-3,7,7-trimethoxynorborn-2-ene (Fig. 1, abbreviated as CMBN). The aim was to obtain information about the anomeric effect in the CH_3—O—C(7)—O—CH_3 moiety and to investigate the influence (if any) of substituents on the distortion of the bicyclo[2.2.1]hept-2-ene skeleton. Previous determinations include the following closely related molecules: 1,2,4-trichloro-3,7,7-trimethoxynorborn-2-ene-5,6-*endo*-dicarboxylic anhydride (Van Hemelrijk, Lenstra & Geise, 1981), 1,2,4-trichloro-3,7,7-trimethoxy-5-methylenenorborn-2-ene (Van Hemelrijk & Lenstra, 1981), 5-*endo*-(bromomethyl)-1,2-*endo*,4-trichloro-7,7-dimethoxynorbornan-3-one (Van de Mieroop & Lenstra, 1978), 6-*endo*-(bromomethyl)-1,2-*endo*,4-trichloro-3-*exo*-(2,2,2-trifluoroethoxy)-3-*endo*,7,7-trimethoxynorbornane (Van de Mieroop, Lenstra & Geise, 1979), and 6-*endo*-(bromomethyl)-1,2-*endo*,4-trichloro-3-*endo*-ethoxy-3,7,7-trimethoxynorbornane (Lenstra, Van de Mieroop, Geise, Van Bree & Anteunis, 1980).

Table 1. *Positional parameters of the molecule in fractions of the cell edges*

E.s.d.'s in parentheses refer to the last digit. H atom (x,j) (with $j = 1, 2, 3$) is attached to C(x). Isotropic temperature factors (Å^2) for non-H atoms are calculated from anisotropic temperature parameters assuming equal volume of the 50% probability region according to Lipson & Cochran (1968): $B_{iso} = 8\pi^2(U^o_{11}U^o_{22}U^o_{33})^{1/3}$. All anisotropic thermal parameters were physically acceptable, except for C(8) and C(10).

	x	y	z	B_{iso}
Br	0·2140 (2)	0·0165 (1)	0·9237 (1)	6·01
Cl(1)	0·4787 (5)	0·3507 (2)	1·1517 (3)	5·16
Cl(2)	0·7618 (4)	0·2068 (3)	1·2490 (3)	4·67
Cl(4)	0·1565 (5)	0·0856 (2)	1·3264 (3)	4·70
C(1)	0·408 (2)	0·2468 (7)	1·1810 (10)	2·71
C(2)	0·552 (1)	0·1924 (8)	1·2481 (9)	2·77
C(3)	0·476 (2)	0·1306 (8)	1·292 (1)	3·14
C(4)	0·280 (1)	0·1413 (8)	1·252 (1)	3·05
C(5)	0·231 (1)	0·1239 (8)	1·120 (1)	3·44
C(6)	0·322 (2)	0·1966 (8)	1·070 (1)	4·07
C(7)	0·274 (1)	0·2378 (8)	1·251 (1)	3·05
O(8)	0·333 (1)	0·2835 (5)	1·3572 (7)	4·00
C(8)	0·235 (2)	0·2800 (10)	1·442 (1)	3·10
O(9)	0·105 (1)	0·2618 (5)	1·1975 (7)	3·65
C(9)	0·067 (2)	0·3523 (8)	1·175 (1)	4·12
C(10)	0·274 (2)	0·0310 (10)	1·092 (1)	3·99
O(11)	0·527 (1)	0·0595 (5)	1·3541 (7)	4·22
C(11)	0·705 (2)	0·0430 (9)	1·383 (1)	5·18
H(5,1)	0·10 (1)	0·116 (6)	1·108 (9)	6·00
H(9,2)	0·12 (1)	0·383 (7)	1·248 (8)	6·00
H(9,3)	0·15 (1)	0·360 (7)	1·141 (8)	6·00
H(8,1)	0·25 (1)	0·341 (7)	1·539 (8)	6·00
H(8,2)	0·08 (1)	0·294 (7)	1·421 (9)	6·00
H(8,3)	0·20 (1)	0·237 (7)	1·474 (8)	6·00
H(6,1)	0·25	0·227	1·019	6·00
H(6,2)	0·40	0·174	1·035	6·00
H(11,1)	0·73	−0·004	1·425	6·00
H(11,2)	0·75	0·039	1·319	6·00
H(11,3)	0·76	0·088	1·425	6·00
H(9,1)	−0·05	0·364	1·144	6·00
H(10,1)	0·39	0·021	1·120	6·00
H(10,2)	0·21	−0·006	1·124	6·00

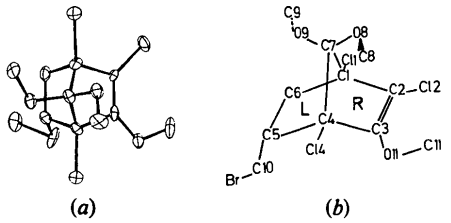


Fig. 1. (*a*) Conformation of the molecule and (*b*) numbering of the atoms.

Intensity data with $\theta \leq 27°$ were collected at room temperature on an Enraf–Nonius CAD-4 diffractometer using Zr-filtered Mo radiation ($\lambda = 0{\cdot}71073$ Å). Control of three reference reflections, measured every 2 h, showed a slight decay of the crystal. The measured intensities were rescaled to account for this decay. No absorption correction ($\mu = 3{\cdot}54$ mm^{-1}) was applied. From the original set of 3156 independent data points, 1478 reflections with $I \geq 2\sigma(I)$ were used in the analysis. The structure was solved applying *MULTAN* (Germain, Main & Woolfson, 1971). About half of the H atoms refined to physically acceptable positions, the others were fixed at their expected positions. In the least-squares refinement all H atoms were given a Debye–Waller temperature parameter fixed at 6·0 Å^2. The weighting scheme used was based on counting statistics. The weighted R value, defined as $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$ over observed reflections, converged to 0·071. An extinction correction did not seem necessary.

The maximum noise level in the final difference electron density map was 0·9 e Å^{-3}. Refined parameters are listed in Table 1 and a view of the molecule is presented in Fig. 1.*

Discussion. Bond distances and valence angles are presented in Table 2 and selected torsion angles are in Table 3.

An important aspect of norbornane and norborn-2-ene geometry is the role substituents play in a possible distortion of the rings. Five-membered rings can be described in terms of the pseudorotation parameters Δ and φ_{max} (Altona, Geise & Romers,

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36897 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and valence angles* (°) *of the title compound*

The standard deviations (×2·5) in parentheses refer to the last digit.

C(1)–C(2)	1·498 (17)	C(4)–Cl(4)	1·721 (12)
C(1)–C(6)	1·548 (17)	C(5)–C(6)	1·542 (17)
C(1)–C(7)	1·518 (17)	C(5)–C(10)	1·545 (20)
C(1)–Cl(1)	1·783 (12)	C(7)–O(8)	1·435 (15)
C(2)=C(3)	1·315 (17)	C(7)–O(9)	1·402 (15)
C(2)–Cl(2)	1·691 (15)	C(8)–O(8)	1·424 (15)
C(3)–C(4)	1·527 (17)	C(9)–O(9)	1·462 (17)
C(3)–O(11)	1·350 (17)	C(10)–Br	1·963 (12)
C(4)–C(5)	1·556 (17)	C(11)–O(11)	1·403 (17)
C(4)–C(7)	1·513 (17)		
C(2)–C(1)–C(6)	107·2 (10)	C(5)–C(4)–Cl(4)	113·6 (10)
C(2)–C(1)–C(7)	101·4 (11)	C(7)–C(4)–Cl(4)	119·2 (10)
C(2)–C(1)–Cl(1)	112·8 (10)	C(4)–C(5)–C(6)	103·4 (11)
C(6)–C(1)–C(7)	101·2 (11)	C(4)–C(5)–C(10)	111·6 (12)
C(6)–C(1)–Cl(1)	113·3 (10)	C(6)–C(5)–C(10)	117·4 (13)
C(7)–C(1)–Cl(1)	119·2 (10)	C(1)–C(6)–C(5)	101·6 (11)
C(1)–C(2)=C(3)	105·4 (12)	C(1)–C(7)–C(4)	94·0 (10)
C(1)–C(2)–Cl(2)	123·7 (11)	C(1)–C(7)–O(8)	108·5 (12)
C(3)=C(2)–Cl(2)	130·3 (12)	C(1)–C(7)–O(9)	117·1 (13)
C(2)=C(3)–C(4)	109·2 (14)	C(4)–C(7)–O(8)	119·2 (12)
C(2)=C(3)–O(11)	135·8 (15)	C(4)–C(7)–O(9)	107·2 (12)
C(4)–C(3)–O(11)	114·7 (14)	O(8)–C(7)–O(9)	110·2 (11)
C(3)–C(4)–C(5)	106·6 (11)	C(5)–C(10)–Br	108·7 (10)
C(3)–C(4)–C(7)	98·1 (12)	C(7)–O(8)–C(8)	119·0 (11)
C(3)–C(4)–Cl(4)	117·0 (11)	C(7)–O(9)–C(9)	118·4 (11)
C(5)–C(4)–C(7)	99·6 (11)	C(3)–O(11)–C(11)	116·4 (12)

Table 3. *Selected torsion angles* (°) *in the title compound*

E.s.d.'s are *ca* 1°; the sign convention is according to IUPAC (1974).

C(6)–C(1)–C(2)=C(3)	−72·9	C(2)=C(3)–O(11)–C(11)	−0·3
C(7)–C(1)–C(2)=C(3)	32·8	C(3)–C(4)–C(5)–C(6)	−64·2
C(2)–C(1)–C(6)–C(5)	70·1	C(7)–C(4)–C(5)–C(6)	37·3
C(7)–C(1)–C(6)–C(5)	−35·7	C(3)–C(4)–C(7)–C(1)	50·2
C(2)–C(1)–C(7)–C(4)	−51·7	C(5)–C(4)–C(7)–C(1)	−58·2
C(2)–C(1)–C(7)–O(8)	70·9	C(5)–C(4)–C(7)–O(9)	61·8
C(6)–C(1)–C(7)–C(4)	58·6	Cl(4)–C(4)–C(7)–C(1)	177·5
Cl(1)–C(1)–C(7)–C(4)	−176·3	C(4)–C(5)–C(6)–C(1)	−0·9
C(1)–C(2)=C(3)–C(4)	1·2	C(10)–C(5)–C(6)–C(1)	−124·5
Cl(2)–C(2)=C(3)–O(11)	2·8	C(6)–C(5)–C(10)–Br	−59·1
C(2)=C(3)–C(4)–C(5)	68·0	O(9)–C(7)–O(8)–C(8)	54·8
C(2)=C(3)–C(4)–C(7)	−34·6	O(8)–C(7)–O(9)–C(9)	54·7

1968). We define $\Delta = 0$ for the envelope (C_s) form having the mirror plane through C(7). The Δ values for the rings are $\Delta_R = -1{\cdot}0°$ and $\Delta_L = 1{\cdot}2°$, revealing that both are close to the ideal C_S form. Also the values $\varphi^R_{max} = 52{\cdot}6°$ and $\varphi^L_{max} = 61{\cdot}8°$ are normal for norborn-2-enes (Van Hemelrijk, Lenstra & Geise, 1981).

Direct comparison of bond lengths and angles with those of a norborn-2-ene reference skeleton (Van Hemelrijk & Lenstra, 1981; Van Hemelrijk, Lenstra & Geise, 1981) shows that at the present level of experimental accuracy the attachment of exocyclic substituents has no systematic effect upon endocyclic geometry.

Three fragments in the structure of CMBN deserve attention. First, in the CH_3–O–C(7)–O–CH_3 moiety, the interaction of the lone pairs at O with antibonding orbitals of the adjacent bonds requires a *gauche,gauche* (*sc,sc*) conformation for maximum orbital overlap. Such a conformation with torsion angles around C(7)–O of about 55° is always observed. As a consequence of the n–σ^* interaction the O(8)–C(7)–O(9) region becomes more sp^2-like. It results on the average in a lengthening of the peripheral C–O bonds [C(9)–O(9) and C(8)–O(8)], a shortening of the central C(7)–O bonds and an opening of the corresponding valence angles. The anomerically induced (*sc,sc*) conformation brings C(8) close to C(4) and C(9) close to C(1). Part of the steric strain is then relieved by letting the angles C(4)–C(7)–O(8) and C(1)–C(7)–O(9) become *ca* 10° larger than C(4)–C(7)–O(9) and C(1)–C(7)–O(8). Similar differences can be observed in all other 7,7-dimethoxy derivatives.

Second, the CH_2Br substituent at C(5) is *endo* with respect to the norbornene system. To the Br atom, or any atom β-situated to the ring system at C(2), C(3), C(5) and C(6), three non-equivalent positions (Y, Y' and Y'' in Fig. 2) are available. In CMBN and the three other compounds with a bromomethyl group we find Br in the Y' position. Other β atoms in the series equally occupy this position unless prevented by distinct physical reasons. At least for CH_2Br-substituted systems, the preferred (*ap*) form is also found (Van Bree & Anteunis, 1981) in solution by ^1H NMR. The (*ap*) form brings the β atom close to a ring C causing again an inequivalence of the exocyclic valence angles involved. For example in the title compound C(10)–

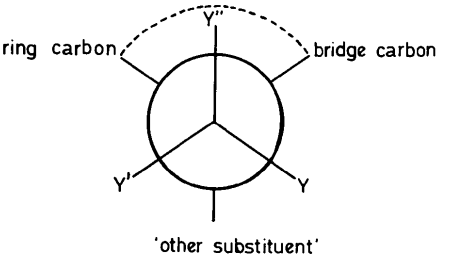
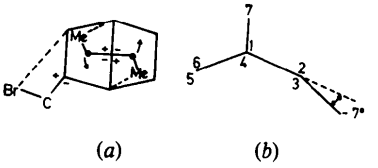


Fig. 2. Projection along a substituent bond attached to *e.g.* C(2). If the 'substituent' is in the *endo*-position the 'other substituent' is in the *exo*-position and *vice versa*. C(1) and C(4) are called bridge atoms, C(2), C(3), C(5) and C(6) are ring atoms. Conformations are described as antiperiplanar (*ap*), synclinal (*sc*), *etc.* according to IUPAC (1974).



(*a*) (*b*)

Fig. 3. (*a*) Top view of a norbornane skeleton showing the effects of the interaction between the skeleton and substituents. (*b*) Side view of a norbornene skeleton substituted at C(2) and C(3).

C(5)–C(6) is larger than C(10)–C(5)–C(4). Similar differences of about 4° are consistently observed throughout the series (*cf.* Fig. 3*a*).

Third, the fragment C(2)=C(3)–O(11)–C(11) has a nearly ideal synperiplanar conformation and agrees with the *syn* conformation observed by Durig & Compton (1978) in gaseous methyl vinyl ether. Again throughout the series, the valence angles Cl(2)–C(2)=C(3) and C(2)=C(3)–O(11) are extremely large, *viz.* 130 and 135° respectively. Although the parts C(1)–C(2)=C(3)–C(4) and Cl(2)–C(2)=C(3)–O(11) are individually planar, the total arrangement around C(2)=C(3) is slightly bent. The plane Cl(2)C(2)=C(3)O(11) is turned away from C(7) and is at an angle of 7° to the C(1)C(2)=C(3)C(4) plane (Fig. 3*b*).

Again it seems that 1–4 interactions, now between the Cl atoms on the bridge C atoms and the opposed substituent on the C(sp^2) ring C atoms, cause this deviation from planarity. Indeed the torsion angles Cl–C(bridge)–C(sp^2)–substituent are about 20–27° in all three norbornenes of the series which is 4–10° more than they would be in an analogous planar configuration around C(2)=C(3).

All these effects together show that the interaction between substituent and ring system affects the substituent geometry and destroys any local symmetry that intuitively might be expected at the junction.

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The Absolute Configuration of Active and Inactive Fosfomycin

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Abstract. $C_3H_6O_3P^-.C_8H_{12}N^+.H_2O$ (inactive), $P2_1$, a = 11·530 (1), b = 6·1490 (3), c = 10·199 (1) Å, β = 102·76 (1)°, Z = 2, D_x = 1·305 Mg m^{-3}. The structure was refined with 1306 observed Friedel pairs to R_{obs} = 0·040 with Cu $K\alpha$ radiation. The X-ray intensities were used to determine the absolute stereochemistry of both enantiomers of fosfomycin. The active form is 1*R*,2*S*, the inactive form being 1*S*,2*R*.

Introduction. Fosfomycin, (–)-(1*R*,2*S*)-(1,2-epoxypropyl)phosphonic acid (Christensen, Leanza, Beattle, Patchett, Arison, Ormond, Kuehl, Albers-Schonberg & Jardetzky, 1969), is now one of the most frequently used antibiotics, because of its effectiveness in inhibiting the growth of various microorganisms (Mastroeni, Nistico, Carbone & Rotiroti, 1980). Experiments have shown that the action of fosfomycin has been identified with the first phase of the bacterial cell-wall synthesis at the stage when the wall component *N*-acetylmuramylpeptide is forming. The enzyme involved is uridinediphospho-*N*-acetylglucosamine-3-*O*-enolpyruvyltransferase, otherwise known as pyruvyltransferase (Kahan, Kahan, Cassidy & Kropp, 1974).

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