indicate some distortion towards the $^5H_6$ half chair with O(1)—C(6)—C(5)—C(4) and O(1)—C(2)—C(3)—C(4) torsion angles 59·7 (3) and −41·6 (3)°, respectively. Similar deformations were observed in carbohydrates with the sp^2-hybridized C atom in position 2 of the pyranoid ring. The distortion in the title compound corresponds to a small deformation of the chair conformation (Ciunik, Szweda & Smiatacz, 1991).

The benzyloxyphenyl group is an axial substitutent (2*a*) of the pyranoid ring and the O(1)—C(2)—C(7)—C(12) torsion angle is −17·0 (3)°. In consequence the pyranoid and the aromatic rings are almost perpendicular to each other. The benzyloxyphenyl substituent has the extended antiperiplanar conformation with C(10)—O(3)—C(13)—C(14) and C(11)—C(10)—O(3)—C(13) torsion angles of −175·6 (2) and −2·9 (3)°, respectively. Both aromatic rings are mutually twisted. All bond lengths and valence angles have normal values.

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Temperature Effect on the Structure of the Complex 1,8-Bis(dimethylamino)naphthalene–Chloranilic Acid[*] (2/1) Dihydrate

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Abstract. $2C_{14}H_{18}N_2.C_6H_2Cl_2O_4.2H_2O$, M_r = 673·64, triclinic, $P\bar{1}$, $Z = 1$, λ(Mo $K\alpha$) = 0·71073 Å, μ = 2·5 cm^{-1}, $F(000) = 356$. Cell dimensions at 150 K: a = 9·3051 (7), b = 9·3261 (4), c = 9·5779 (8) Å, α = 104·604 (5), β = 98·486 (6), γ = 92·913 (5)°, V = 792·1 (1) Å^3, D_x = 1·412 g cm^{-3}, R = 0·040 for 3343 reflections with $I \geq 2{\cdot}5\sigma(I)$. At 295 K: a = 9·420 (3), b = 9·444 (3), c = 9·626 (3) Å, α = 104·84 (4), β = 97·84 (4), γ = 92·66 (4)°, V = 817·1 (5) Å^3, D_x = 1·369 g cm^{-3}, R = 0·036 for 2426 reflections with $I \geq 3{\cdot}0\sigma(I)$. The structure consists of centrosymmetric doubly ionized chloranilic acid anions and protonated bis(dimethylamino)naphthalene cations. The water molecules act as cohesive elements by connecting the anions through hydrogen bonds to form endless chains which run in the **a** direction. The channels formed by these chains accommodate pairs of centrosymmetrically related cations. The intramolecular NHN$^+$ hydrogen bonds in the cation with distances of 2·589 (3) (at 295 K) and 2·588 (2) Å (at 150 K) are asymmetric and nonlinear. The asymmetry, which is caused by the interaction with the chloranilate anion, increases on cooling.

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[*] 2,5-Dichloro-3,6-dihydroxy-*p*-benzoquinone.

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Introduction. Structural studies on the protonated 'proton sponge' 1,8-bis(dimethylamino)naphthalene (DMAN) performed for various salts (Truter & Vickery, 1972, Pyżalska, Pyżalski & Borowiak, 1983; Głowiak, Malarski, Sobczyk & Grech, 1983; Malarski, Lis, Grech, Nowicka-Scheibe & Majewska, 1990; Woźniak Krygowski, Kariuki, Jones & Grech, 1990; Kanters, Schouten, Kroon & Grech, 1991) indicate that strong intramolecular NHN$^+$ hydrogen bonds of 2·554 (5) to 2·65 (2) Å are formed. The arrangement of the proton with respect to the two N atoms can be symmetric or asymmetric, possibly depending on the lattice potential in the neighbourhood of the bridge. The bridge asymmetry is also reflected in other bond lengths of the DMAN.H$^+$ cation. It seemed interesting to perform studies of the temperature effect on the structure of the DMAN.H$^+$ fragment. It has been shown (Grech, Malarski & Sobczyk, 1978; Grech, Malarski, Romanowski & Sobczyk, 1978; Grech, Malarski, Ilczyszyn, Czupiński, Sobczyk, Rozière, Bonnet & Potier, 1985; Grech, Malarski, Sobczyk, Potier & Rozière, 1988; Baran, Malarski, Sobczyk & Grech, 1988) that temperature has a large influence on the IR spectra of homoconjugated NHN$^+$ cations, producing considerable red shift of the high frequency $\nu_{\mathrm{N—H\cdots N}}$ band and an increase of intensity of the absorption at about 500 cm^{-1}. The temperature effect on the structure of the NHN$^+$ bridge has only been studied for quinuclidin-3-one hemiperchlorate (Rozière, Belin & Lehman, 1982). At 120 K the NHN$^+$ bridge is symmetrical, while at room temperature two minima of equal energy for the proton motion should be located above and below the line linking the N atoms (Jones, Brach & Rozière, 1984).

The subject of the present study is the dihydrated 2:1 complex of DMAN with chloranilic acid. The compound crystallized in the form of protonated DMAN and doubly ionized chloranilic acid. Because the room-temperature X-ray diffraction studies showed an asymmetric NHN$^+$ bridge, we decided to extend the studies to low temperature (150 K). The IR spectra of the complex are characteristic for DMAN.H$^+$ with an elongated NHN$^+$ bridge. In addition to the main ν_s(NHN) band at about 500 cm^{-1}, a weak broad absorption centred at about 2000 cm^{-1} is observed.

Experimental. In the following, data for the room-temperature analysis are given in square brackets. A red crystal of dimensions 0·8 × 0·3 × 0·2 [0·75 × 0·35 × 0·25 mm] was used for data collection on an Enraf–Nonius CAD-4 [Syntex $P2_1$] diffractometer with Zr-filtered [graphite monochromator] Mo $K\alpha$ radiation. Lattice parameters were determined from the setting angles of 25 [15] reflections in the range $9{\cdot}6 < \theta < 13{\cdot}9$ [$18{\cdot}0 < \theta < 20{\cdot}0°$]. The diffracted intensities of 3617 [2700] unique reflections, $R_{\mathrm{int}} = 0{\cdot}023$ [0·034], were collected using the ω–2θ scan mode, $2\theta_{\max} = 55$ [49°], of which 3343 [2426] were above the $2{\cdot}5\sigma(I)$ [$3{\cdot}0\sigma(I)$] level. h −12 to 11, k −12 to 11, l 0 to 12 [h 0 to 11, k −11 to 11, l −11 to 11]. Two periodically measured standard reflections (516, 423) [232, 331], measured every hour, showed an r.m.s. deviation of 2·4 [6·0%] during data collection. Intensities were corrected for Lp effects, but not for absorption. The choice of the space group $P\overline{1}$ was later confirmed by the course of the refinement. The structure was solved by direct methods with *SHELXS*86 (Sheldrick, 1986). The H atoms were located on difference Fourier maps and included in the refinement with individual isotropic thermal parameters. Anisotropic full-matrix least-squares refinement on F of 292 parameters converged at $R = 0{\cdot}040$ [0·036] and $wR = 0{\cdot}054$ [0·043] with $w = 1/\sigma^2(F_o)$, $S = 0{\cdot}49$ [1·17], $(\Delta/\sigma)_{\mathrm{av}} = 0{\cdot}023$ [0·01] and $(\Delta/\sigma)_{\max} = 0{\cdot}63$ [0·10]. Maximum and minimum densities in the final difference map were 0·64 and −0·28 [0·18 and −0·18 e Å^{-3}], respectively. The greater part of the positive difference electron density in the low-temperature study is located on the middle of the bonds between non-H atoms. The scattering factors were those of Cromer & Mann (1968) and anomalous-dispersion terms were from Cromer & Liberman (1970). Calculations were performed with *SHELX*76 (Sheldrick, 1976), *CXTL/XTLE* (Syntex, 1976), for the refinement, and the *EUCLID* (Spek, 1982) package for the geometry and illustrations, on a MicroVAXII [IBM PC-AT] computer.

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1.* Bond distances and angles involving non-H atoms are collected in Table 2.

The asymmetric unit consists of one protonated DMAN molecule, one half chloranilate anion and one water molecule. The anion lies at an inversion centre. The water molecule connects two chloranilate anions which are one translation apart along **a** by means of a two- and a three-centre hydrogen bond, respectively. One water proton donates to an O(2) atom of the anion and the other proton donates to two neighbouring O(1) and O(2) atoms of the anion at $(1 + x, y, z)$ and $(1 - x, 2 - y, -z)$ respectively (see Fig. 1). Thus the crystal contains endless chains of anions along **a** linked by water molecules and these chains encapsulate pairs of centrosymmetrically

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* 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 54093 (40 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final coordinates and equivalent isotropic thermal parameters* (Å^2) *at* 150 K (*first value*) *and* 295 K (*second value*)

$$U_{\mathrm{eq}} = (1/3)\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq}
Cl(1)	0·18045 (4)	0·81825 (4)	0·19277 (4)	0·0193 (1)
	0·1777 (1)	0·8236 (1)	0·1932 (1)	0·0586 (3)
O(1)	−0·1421 (1)	0·8207 (1)	0·1269 (1)	0·0223 (3)
	−0·1396 (2)	0·8275 (2)	0·1304 (2)	0·0680 (10)
O(2)	0·2960 (1)	1·0200 (1)	0·0296 (1)	0·0174 (3)
	0·2914 (2)	1·0187 (2)	0·0281 (2)	0·0505 (8)
O(3)	0·5146 (1)	0·8217 (1)	0·1094 (1)	0·0221 (3)
	0·5156 (3)	0·8234 (2)	0·1090 (3)	0·0678 (11)
N(1)	0·1975 (1)	0·4872 (1)	0·7764 (1)	0·0133 (3)
	0·1965 (2)	0·4849 (2)	0·7744 (2)	0·0371 (8)
N(2)	0·1743 (1)	0·2578 (1)	0·5534 (1)	0·0129 (3)
	0·1775 (2)	0·2592 (2)	0·5512 (2)	0·0354 (8)
C(1)	0·2571 (2)	0·5771 (2)	0·6886 (2)	0·0121 (3)
	0·2549 (2)	0·5754 (2)	0·6880 (2)	0·0335 (9)
C(2)	0·2969 (2)	0·7257 (2)	0·7496 (2)	0·0156 (4)
	0·2920 (2)	0·7216 (2)	0·7488 (2)	0·0436 (10)
C(3)	0·3546 (2)	0·8135 (2)	0·6677 (2)	0·0174 (4)
	0·3478 (3)	0·8093 (3)	0·6678 (3)	0·0511 (12)
C(4)	0·3714 (2)	0·7503 (2)	0·5264 (2)	0·0165 (4)
	0·3659 (2)	0·7479 (3)	0·5281 (3)	0·0466 (11)
C(5)	0·3347 (2)	0·5959 (2)	0·4611 (2)	0·0132 (3)
	0·3319 (2)	0·5958 (2)	0·4622 (2)	0·0375 (9)
C(6)	0·3604 (2)	0·5295 (2)	0·3174 (2)	0·0165 (4)
	0·3578 (3)	0·5310 (3)	0·3189 (2)	0·0471 (11)
C(7)	0·3317 (2)	0·3793 (2)	0·2569 (2)	0·0172 (4)
	0·3314 (3)	0·3841 (3)	0·2585 (3)	0·0508 (12)
C(8)	0·2713 (2)	0·2896 (2)	0·3359 (2)	0·0153 (4)
	0·2731 (3)	0·2946 (3)	0·3355 (2)	0·0432 (10)
C(9)	0·2414 (2)	0·3498 (2)	0·4735 (2)	0·0124 (3)
	0·2435 (2)	0·3520 (2)	0·4726 (2)	0·0333 (9)
C(10)	0·2751 (1)	0·5053 (2)	0·5427 (2)	0·0114 (3)
	0·2741 (2)	0·5054 (2)	0·5427 (2)	0·0314 (8)
C(11)	0·3037 (2)	0·4864 (2)	0·9094 (2)	0·0186 (4)
	0·3002 (3)	0·4833 (3)	0·9054 (3)	0·0558 (13)
C(12)	0·0535 (2)	0·5313 (2)	0·8134 (2)	0·0192 (4)
	0·0529 (3)	0·5267 (3)	0·8116 (3)	0·0566 (13)
C(13)	0·2581 (2)	0·1314 (2)	0·5714 (2)	0·0181 (4)
	0·2621 (3)	0·1352 (3)	0·5687 (3)	0·0530 (13)
C(14)	0·0212 (2)	0·2062 (2)	0·4859 (2)	0·0187 (4)
	0·0269 (3)	0·2071 (3)	0·4853 (3)	0·0543 (12)
C(15)	0·0817 (2)	0·9185 (2)	0·0864 (2)	0·0152 (4)
	0·0798 (2)	0·9212 (2)	0·0867 (2)	0·0415 (10)
C(16)	−0·0720 (2)	0·9021 (2)	0·0718 (2)	0·0147 (4)
	−0·0706 (2)	0·9060 (2)	0·0737 (2)	0·0425 (10)
C(17)	0·1603 (2)	1·0073 (2)	0·0201 (2)	0·0140 (4)
	0·1576 (2)	1·0068 (2)	0·0189 (2)	0·0390 (10)

related $\mathrm{DMAN.H^+}$ residues near the centre of the unit cells.

The planes of the chloranilic acid anions are parallel, whereas the $\mathrm{DMAN.H^+}$ fragments are 'antiparallel' with a distance between the naphthalene planes of 3·460 (2) and 3·550 (2) Å, at 150 and 295 K, respectively. The angle between the $\mathrm{DMAN.H^+}$ and chloranilate planes is about 90°. The packing of the molecules is shown in the stereoplot of Fig. 2.

At 295 K the $\mathrm{NHN^+}$ hydrogen bond in the cation is asymmetric, although the proton is markedly shifted to the centre of the bridge as follows from the N(1)—H distance of 1·14 (3) Å. The asymmetry is also reflected in the N—C distances, N(2)—C(9) 1·465 (3) and N(1)—C(1) 1·474 (3) Å. The N—H···N hydrogen-bond angle [155 (2)°] deviates considerably from linearity. The N(1) atom is located in the plane of the naphthalene ring, but N(2) is 0·088 (4) Å out of this plane.

Table 2. *Bond distances* (Å) *and bond angles* (°) *at* 150 K (*first value*) *and* 295 K (*second value*)

Cl(1)—C(15)	1·746 (2)	N(2)—C(14)	1·477 (2)
	1·748 (2)		1·479 (3)
C(6)—C(7)	1·370 (2)	O(1)—C(16)	1·238 (2)
	1·357 (2)		1·237 (3)
C(1)—C(2)	1·369 (2)	C(7)—C(8)	1·408 (2)
	1·363 (3)		1·398 (4)
O(2)—C(17)	1·250 (2)	C(1)—C(10)	1·428 (2)
	1·249 (3)		1·425 (3)
C(8)—C(9)	1·371 (2)	N(1)—C(1)	1·471 (2)
	1·364 (3)		1·474 (3)
C(2)—C(3)	1·407 (2)	C(9)—C(10)	1·431 (2)
	1·404 (3)		1·430 (3)
N(1)—C(11)	1·495 (2)	C(3)—C(4)	1·368 (2)
	1·490 (3)		1·359 (4)
C(15)—C(16)	1·414 (2)	N(1)—C(12)	1·488 (2)
	1·403 (3)		1·491 (3)
C(4)—C(5)	1·419 (2)	C(15)—C(17)	1·401 (2)
	1·416 (3)		1·401 (3)
N(2)—C(9)	1·459 (2)	C(5)—C(6)	1·421 (2)
	1·465 (3)		1·417 (3)
C(16)—C(17$^{\mathrm{i}}$)	1·548 (2)	N(2)—C(13)	1·478 (2)
	1·544 (3)		1·478 (3)
C(5)—C(10)	1·430 (2)		
	1·428 (3)		
C(1)—N(1)—C(11)	112·0 (1)	C(7)—C(8)—C(9)	121·1 (1)
	112·1 (2)		121·1 (2)
C(1)—N(1)—C(12)	112·4 (1)	N(2)—C(9)—C(8)	121·2 (1)
	112·4 (2)		121·1 (2)
C(11)—N(1)—C(12)	111·9 (1)	N(2)—C(9)—C(10)	118·1 (1)
	112·1 (2)		118·1 (2)
C(9)—N(2)—C(13)	113·1 (1)	C(8)—C(9)—C(10)	120·7 (1)
	113·2 (2)		120·8 (2)
C(9)—N(2)—C(14)	111·3 (1)	C(1)—C(10)—C(5)	117·1 (1)
	111·8 (2)		117·1 (2)
C(13)—N(2)—C(14)	111·0 (1)	C(1)—C(10)—C(9)	125·1 (1)
	111·2 (2)		125·4 (2)
N(1)—C(1)—C(2)	119·4 (1)	C(5)—C(10)—C(9)	117·8 (1)
	119·8 (2)		117·5 (2)
N(1)—C(1)—C(10)	118·6 (1)	Cl(1)—C(15)—C(16)	117·3 (1)
	118·4 (2)		117·3 (2)
C(2)—C(1)—C(10)	121·9 (1)	Cl(1)—C(15)—C(17)	117·8 (1)
	121·8 (2)		117·4 (2)
C(1)—C(2)—C(3)	120·3 (1)	C(16)—C(15)—C(17)	124·9 (1)
	120·4 (2)		125·0 (2)
C(2)—C(3)—C(4)	120·0 (1)	O(1)—C(16)—C(15)	125·1 (1)
	119·9 (2)		125·1 (2)
C(3)—C(4)—C(5)	121·1 (1)	O(1)—C(16)—C(17$^{\mathrm{i}}$)	117·2 (1)
	121·4 (2)		117·2 (2)
C(4)—C(5)—C(6)	120·8 (1)	C(15)—C(16)—C(17$^{\mathrm{i}}$)	117·7 (1)
	121·1 (2)		117·8 (2)
C(4)—C(5)—C(10)	119·6 (1)	O(2)—C(17)—C(15)	125·4 (1)
	119·4 (2)		125·2 (2)
C(6)—C(5)—C(10)	119·6 (1)	O(2)—C(17)—C(16$^{\mathrm{i}}$)	117·2 (1)
	119·4 (2)		117·6 (2)
C(5)—C(6)—C(7)	120·9 (1)	C(15)—C(17)—C(16$^{\mathrm{i}}$)	117·4 (1)
	121·0 (2)		117·2 (2)
C(6)—C(7)—C(8)	119·8 (1)		
	120·1 (2)		

Symmetry code: (i) $-x$, $2-y$, $-z$.

The cooling from 295 to 150 K does not lead to marked changes in the structure of the chloranilate anion and naphthalene ring. On the other hand considerable deformations occur within fragments embracing the hydrogen bonds (Table 3). The O(3)···O(2) distance changes from 3·019 (3) to 2·962 (2) Å, O(3)···O(2)($1-x$, $2-y$, $-z$) from 2·946 (3) to 2·912 (2) Å and O(3)···O(1)($1+x$, y, z) from 3·224 (3) to 3·175 (2) Å. One of the O(3)—H water donors forms a three-centre hydrogen bond with O(1)($1+x$, y, z) and O(2)($1-x$, $2-y$, $-z$) of the anion with angles of 138 (2) and 150 (3)° at 150 K and 151 (4) and 142 (4)° at 295 K. The four-atom arrangement of these hydrogen bonds is planar, as follows from the sum of angles around the central H atom [360 (4)° at both temperatures]. It is remarkable that the hydrogen bonds between water and the charged chloranilate O atoms are weak, as is indicated by the relatively long distances. Next to these hydrogen bonds there are three C—H···O hydrogen bonds (Table 3) which all show appreciable shortening of the donor···acceptor distances upon cooling.

In contrast to the shortening of the O···O and C···O hydrogen-bond contacts, the N(1)···N(2) distances in the NHN^+ hydrogen bond do not change with temperature [2·589 (3) and 2·588 (2) Å at 295 and 150 K] and the same holds for the hydrogen-bond angles [155 (2) and 152 (2)°]. This is very probably a result of the very short and limiting intramolecular N···N distance. However, the bridge becomes more asymmetric upon cooling, the N(1)—H distance decreases from 1·14 (3) to 1·07 (3) Å. This temperature effect may be related to the change of the mutual arrangement of the NHN^+ bridge and the chloranilate anions. The distances listed in Table 3 clearly show the enhancement of interaction upon cooling between O(1) and the NHN^+ fragment. O(1), which is located closer to N(1) than to N(2), is forced into a more asymmetric position with respect to the two N atoms upon cooling.

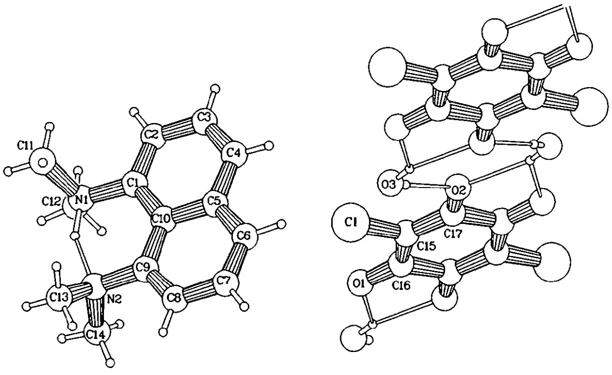

Fig. 1. Protonated DMAN and the hydrated chloranilate anion chain with atom numbering.

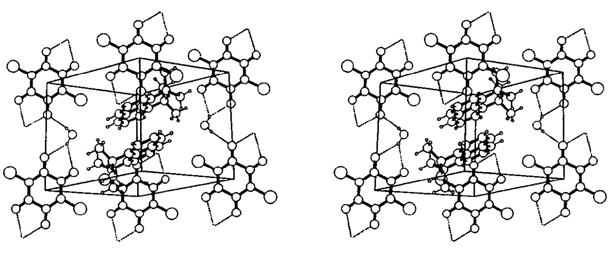

Fig. 2. Stereoview of the crystal packing.

Table 3. *Geometry of* N—H···N, O—H···O *and* C—H···O *hydrogen bonds and intermolecular contact distances between non-*H *atoms of the cation and anion at* 150 K (*first value*) *and* 295 K (*second value*)

Donor Acceptor	*D*···*A* (Å)	*D*—H (Å)	H···*A* (Å)	*D*—H···*A* (°)
$N(1)—H\cdots N(2^{i})$	2·588 (2)	1·07 (3)	1·59 (2)	152 (2)
	2·589 (3)	1·14 (3)	1·51 (3)	155 (2)
$O(3)—H\cdots O(1^{ii})$	3·175 (2)	0·89 (3)	2·46 (3)	138 (2)
	3·224 (3)	0·77 (4)	2·53 (4)	151 (4)
$O(3)—H\cdots O(2^{iii})$	2·912 (2)	0·89 (3)	2·11 (3)	150 (3)
	2·946 (3)	0·77 (4)	2·30 (4)	142 (4)
$O(3)—H'\cdots O(2^{i})$	2·962 (2)	0·80 (3)	2·17 (3)	171 (3)
	3·019 (3)	0·77 (4)	2·26 (4)	169 (4)
$C(2)—H\cdots O(2^{iv})$	3·320 (2)	0·96 (2)	2·49 (2)	145 (2)
	3·353 (3)	0·97 (3)	2·55 (3)	140 (2)
$C(11)—H\cdots O(1^{v})$	3·081 (2)	0·98 (2)	2·41 (2)	125 (1)
	3·154 (4)	0·96 (3)	2·44 (3)	131 (2)
$C(13)—H\cdots O(1^{v})$	3·167 (2)	0·95 (2)	2·47 (2)	131 (2)
	3·205 (4)	0·99 (3)	2·49 (3)	129 (2)

$O(1)\cdots N(1^{v})$	3·272 (2)	$O(1)\cdots C(12^{v})$	3·587 (2)
	3·347 (3)		3·641 (4)
$O(1)\cdots N(2^{v})$	3·374 (2)	$O(1)\cdots C(14^{v})$	3·788 (2)
	3·427 (3)		3·808 (4)

Symmetry code: (i) *x*, *y*, *z*; (ii) 1 + *x*, *y*, *z*; (iii) 1 − *x*, 2 − *y*, −*z*; (iv) *x*, *y*, 1 + *z*; (v) −*x*, 1 − *y*, 1 − *z*.

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