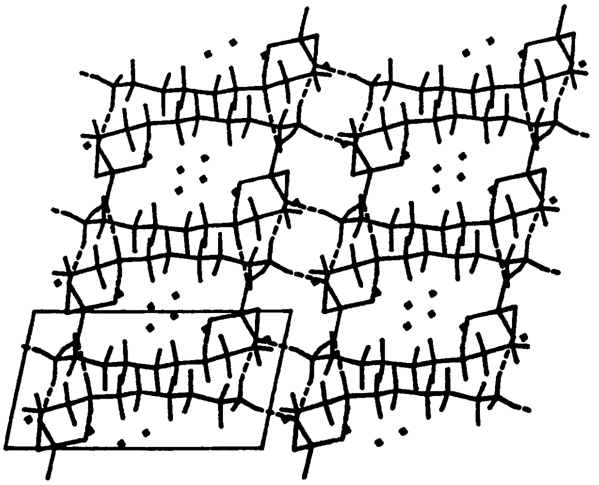

Fig. 3. Packing diagram of WR 2823 dihydrate viewed down the *b* axis. The intramolecular hydrogen bond and hydrogen bonds between WR 2823 molecules are depicted by the dotted lines. The small crosses represent the location of the O atoms of the water molecules *W*(1) to *W*(4). The hydrogen bonds involving the water molecules are not illustrated. The figure was drawn using *SYBYL* (Tripos Associates, 1989).

cannot be present simultaneously since they would be only 1·71 (1) Å apart. The WR 2823 molecules pack such that the hydrophobic sides of the molecules face each other and the hydrophilic sides of the molecules face the water channels.

In summary, from studying this series of radioprotective agents, we have discovered that the S—P bond length is longer than usual, but of consistent length. These molecules all crystallized as zwitterions in the presence of two or three molecules of water per radioprotectant. This results in P—O bonds of essentially equal length. An intramolecular hydrogen bond forms when $n' = 2$, although the *gauche*-staggered nature of the —S—CH$_2$—CH$_2$—N— segment of the molecule is not dependent upon this hydrogen bond. Finally, the linear or bent nature of the molecule is apparently dependent on the numerical values of n and n'.

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A Novel Aminotriol: 4-Amino-4-(3-hydroxy-1-propyl)-1,7-heptanediol

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Abstract. $C_{10}H_{23}NO_3$, $M_r = 205{\cdot}30$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}314\,(2)$, $b = 9{\cdot}539\,(1)$, $c = 14{\cdot}512\,(3)$ Å, $V = 1150{\cdot}9\,(6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}185$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}8$ cm^{-1}, $F(000) = 456$, $T = 293\,(2)$ K, 1926 unique reflections measured, final $R = 0{\cdot}034$ for 1681 reflections with I

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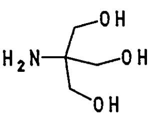
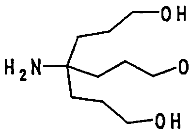
$> 3{\cdot}0\sigma(I)$. The amino group and all three hydroxyl groups are involved in ten intermolecular hydrogen bonds, ranging in length from 2·776 (2) to 3·314 (2) Å, resulting in an unexpectedly high melting point (382 K). The C—N bond length is 1·483 (2) Å, and the mean C—O bond length is 1·420 (6) Å. The C—C bond distances of the hydroxypropyl groups exhibit shortening with increasing distance from the central carbon atom, with means of 1·541 (2), 1·523 (4) and 1·514 (6) Å.

Introduction. Our continuing interest in cascade polymers (Buhleier, Wehner & Vögtle, 1978; Newkome, Yao, Baker & Gupta, 1985; Newkome, Baker, Saunders, Russo, Gupta, Yao, Miller & Bullion, 1986), which we envisioned could be synthesized *via* an iterative method involving (1) ester amidation with a multifunctional amine such as tris(hydroxymethyl)aminomethane [Tris, (I)], (2) transformation of the hydroxyl moieties to better leaving groups and (3) alkylation with an ester (a trialkyl methanetricarboxylate), has led to the design and synthesis of a new synthon, 4-amino-4-(3-hydroxy-1-propyl)-1,7-heptanediol [Bis-homotris, (II)] (Newkome, Moorefield & Theriot, 1988). Realization of the multiplicative procedure was precluded by alkylation with a bulky nucleophile at a neopentyl carbon. It was therefore surmised that a one-carbon homologation of the hydroxyl groups could circumvent the problem; however, our synthetic results indicated and confirmed (Rice, Sheth & Zalucky, 1971) that at least a two-carbon homologation was necessary. As the functional equivalent of Tris, Bis-homotris is characterized by a two-carbon homologation of each hydroxyl group from the quaternary core. Crystal structure determination of the new aminotriol would afford key insight into the nature of hydrogen bonding (intramolecular *versus* intermolecular) and hence chemical reactivity.



(I)



(II)

Experimental. A colorless prismatic crystal (0·30 × 0·35 × 0·50 mm) of (II) was mounted on a glass fiber, in random orientation, on an Enraf–Nonius CAD-4 κ-axis diffractometer equipped with a graphite-crystal incident-beam monochromator. Cell constants were obtained from least-squares refinement using 25 reflections in the range of $11 < \theta < 13°$. From the systematic absences ($h00$, h odd; $0k0$, k odd; and $00l$, l odd) the space group was determined to be $P2_12_12_1$. Data were collected *via* the ω–2θ scan mode, with a variable scan rate of 1–3° min^{-1} ($h = 0$ to 11, $k = 0$ to 13, $l = 0$ to 20) to a maximum 2θ of 60°. A total of 2018 reflections was measured, 1926 of which were unique. Standard reflections 400, 020 and 004 were measured every 10000 s as a check of crystal and electronic stability and showed a total intensity loss of 1·5%, and a linear decay correction was applied. Lorentz and polarization corrections were applied. No absorption correction was made.

The structure was solved by direct methods; all non-hydrogen atoms were located from an E map. Hydrogen-atom coordinates were obtained from difference Fourier syntheses. The model was refined by full-matrix least squares; all non-hydrogen atoms were refined anisotropically and all hydrogen atoms were refined isotropically. The function minimized was $\sum w(|F_o| - |F_c|)^2$ and the weighting function used was $4F_o\text{Lp}/[S^2(C + R^2B) + (pF_o^2)^2]$ where the value of p was set at 0·020, S = scan rate, C = integrated count, R = scan time/background time, and B = background count.

Scattering factors, including anomalous-dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Refinement used 1681 reflections having intensities greater than three times their standard deviations with 220 variables, converging (largest shift/e.s.d. = 0·05) to $R = 0{\cdot}034$, $wR = 0{\cdot}042$, $R(\text{all}) = 0{\cdot}046$ and $S = 1{\cdot}60$. An extinction coefficient refined to $g = 9\,(2) \times 10^{-7}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. No correlation coefficients were greater than 0·50. The final difference Fourier yielded 0·21 e Å^{-3} for the maximum positive value, while the minimum negative value was −0·14 e Å^{-3}. Programs used were *MULTAN*82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), *SDP*/*VAX* (Frenz, 1978), *ORTEP* (Johnson, 1965) and *PLUTO*78 (Motherwell & Clegg, 1978).

Discussion. Final positional parameters are given in Table 1,* and selected bond lengths and bond angles are shown in Table 2. The numbering scheme and thermal ellipsoids are illustrated in Fig. 1. The C—N and average C—O bond lengths of 1·483 (2) and 1·420 (6) Å, respectively, compare favorably with known values, especially those reported for Tris (Lin, Su, Li & Huang, 1987). The C—C bonds adjacent (α) to the C—O bonds are significantly shorter [average 1·514 (5) Å] than those γ to the C—O

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* Lists of H-atom coordinates and thermal parameters, torsion angles, anisotropic thermal parameters, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53683 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional and equivalent isotropic thermal parameters and their e.s.d.'s*

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

	x	y	z	B_{eq}(Å^2)
O1	1·0983 (2)	0·4513 (2)	0·78303 (7)	3·96 (3)
O2	0·3200 (1)	0·4172 (1)	0·44860 (7)	2·92 (2)
O3	1·1547 (1)	0·7295 (1)	0·22338 (7)	3·31 (2)
N1	0·9285 (1)	0·3528 (1)	0·44325 (7)	2·00 (2)
C1	0·8432 (2)	0·4836 (1)	0·46906 (8)	1·67 (2)
C2	0·9220 (2)	0·5566 (2)	0·55254 (9)	2·06 (2)
C3	0·9614 (2)	0·4630 (2)	0·6346 (1)	2·98 (3)
C4	1·0449 (2)	0·5418 (2)	0·7109 (1)	3·24 (3)
C5	0·8453 (2)	0·5818 (1)	0·38495 (9)	2·12 (2)
C6	1·0097 (2)	0·6047 (2)	0·3405 (1)	2·32 (3)
C7	1·0009 (2)	0·7099 (2)	0·2627 (1)	3·21 (3)
C8	0·6688 (2)	0·4391 (1)	0·48981 (9)	1·92 (2)
C9	0·5492 (2)	0·5579 (2)	0·5029 (1)	2·64 (3)
C10	0·3778 (2)	0·5074 (2)	0·5188 (1)	2·65 (3)

Table 2. *Bond distances* (Å) *and bond angles* (°)

O1—C4	1·427 (2)	C2—C3	1·524 (2)
O2—C10	1·417 (2)	C3—C4	1·508 (2)
O3—C7	1·413 (2)	C5—C6	1·527 (2)
N1—C1	1·483 (2)	C6—C7	1·512 (2)
C1—C2	1·543 (2)	C8—C9	1·519 (2)
C1—C5	1·539 (2)	C9—C10	1·521 (2)
C1—C8	1·541 (2)		
N1—C1—C2	112·0 (1)	O1—C4—C3	112·3 (1)
N1—C1—C5	107·9 (1)	C1—C5—C6	115·6 (1)
N1—C1—C8	105·5 (1)	C5—C6—C7	111·5 (1)
C2—C1—C5	110·0 (1)	O3—C7—C6	110·2 (1)
C2—C1—C8	111·7 (1)	C1—C8—C9	115·8 (1)
C5—C1—C8	109·5 (1)	C8—C9—C10	113·3 (1)
C1—C2—C3	116·1 (1)	O2—C10—C9	113·7 (1)
C2—C3—C4	112·4 (1)		

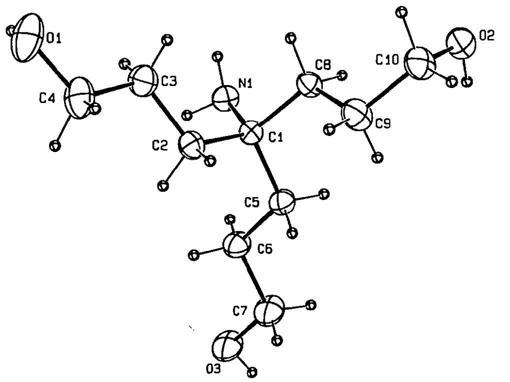

Fig. 1. Thermal ellipsoids drawn at the 50% probability level.

bonds [average 1·541 (2) Å], while those β to the C—O bonds are intermediate [average 1·523 (4) Å]. The α-C—C distance is consistent with that found in Tris [1·519 (3) Å; Lin, Su, Li & Huang (1987); THXMAM02 in the Cambridge Crystallographic Data Files (Allen, Kennard & Taylor, 1983)] and in the hydrogen iodide salt of Tris [1·512 (19) Å; Rudman, Lippman, Sake Gowda & Eilerman (1983)]. The orthorhombic phase of Tris has been studied at six temperatures by Eilerman & Rudman (1980), who found the C—C distance at 295 K to average 1·526 (3) Å.

Table 3. *Hydrogen-bond geometry* (Å, °)

A	*A*—H	H	H···*B*	*B*	*A*H*B*	*A*···*B*
N1	0·86 (2)	HN1	2·33 (2)	O2^{i}	160 (2)	3·148 (2)
N1	0·87 (2)	HN2	2·45 (2)	O2^{ii}	175 (2)	3·314 (2)
O1	0·88 (2)	HO1	1·94 (2)	O3^{iii}	171 (2)	2·818 (2)
O2	0·84 (2)	HO2	1·96 (2)	O1^{iv}	178 (2)	2·794 (2)
O3	0·82 (2)	HO3	1·96 (2)	N1^{v}	176 (2)	2·776 (2)

Symmetry transformations (relative to Table 1) are: (i) $0·5 + x$, $0·5 - y$, $1·0 - z$; (ii) $1·0 + x$, y, z; (iii) $2·5 - x$, $1·0 - y$, $0·5 + z$; (iv) $1·5 - x$, $1·0 - y$, $-0·5 + z$; (v) $2·0 - x$, $0·5 + y$, $0·5 - z$.

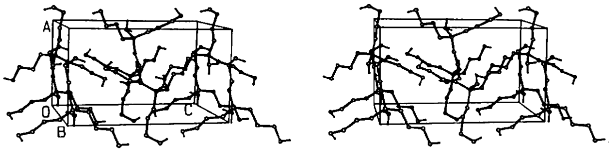

Fig. 2. Stereoview of the unit cell, viewed slightly oblique to the *b* axis.

The amino and three hydroxyl groups form five donor and five acceptor H bonds to ten separate adjacent molecules. This network is described in Table 3 and illustrated in Fig. 2. The amino hydrogen atoms form significantly longer interactions [2·33 (2)–2·45 (2) Å] than the hydroxyl hydrogen atoms [1·94 (2)–1·96 (2) Å].

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