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Organic Hydroxylamine Derivatives. XI.* Structural Analogues of γ-Aminobutyric Acid (GABA) of the Isoxazole Enol-betaine Type. Synthesis and the Crystal Structure of 3-Hydroxy-5-(3-aminopropyl)isoxazole Zwitterion

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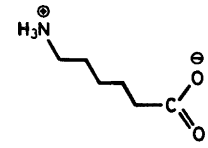
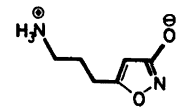
The synthesis and the crystal structure determination of 3-hydroxy-5-(3-aminopropyl)isoxazole zwitterion (V), which is a structural analogue of ε-aminocaproic acid, are described. The synthesis is based on methyl 3-(3-methoxyisoxazol-5-yl)propionate (I), which by conventional methods is converted into (V). The pK_A values of 3-hydroxy-5-(3-aminopropyl)isoxazole zwitterion have been determined to 5.37 ± 0.04 and 10.36 ± 0.04.

Crystals of 3-hydroxy-5-(3-aminopropyl)isoxazole monohydrate, $C_6H_{10}N_2O_2.H_2O$, are monoclinic, space group $C2/c$, $a = 23.45(1)$, $b = 5.768(1)$, $c = 13.483(4)$ Å, $\beta = 120.33(3)°$, $Z = 8$. The structure has been solved by a direct phasing technique using X-ray diffraction data, and has been refined by full-matrix least-squares methods. The final R value is 0.039 for 1218 independent observations. The molecule is a zwitterion, and the crystal structure is stabilized by a system of hydrogen bonds.

As part of our investigations of isoxazole enol-betaines, which are structural analogues of γ-aminobutyric acid (GABA) or closely related amino acids with GABA-like activity in the mammalian central nervous system, the crystal structure determinations of muscimol (3-hydroxy-5-aminomethylisoxazole zwitterion) [1,2] and homomuscimol (3-hydroxy-5-(2-aminoethyl)isoxazole zwitterion) [3] have been performed. The present paper describes the synthesis and the crystal structure determination of 3-hydroxy-5-(3-aminopropyl)isoxazole zwitterion (V), which is a structural analogue of ε-aminocaproic acid.[4] This synthetic amino acid exhibits a weak, GABA-like depressant activity when applied to feline spinal interneurones.[5] The biological properties of ε-aminocaproic acid are, however, multifarious, and rather conflicting results have been obtained after administration of this compound to different structures of the mammalian central nervous system (see Ref. 6). Investigations of the biological properties of 3-hydroxy-5-(3-aminopropyl)isoxazole will be initiated in the near future.



ε-Aminocaproic acid



3-Hydroxy-5-(3-aminopropyl)isoxazole zwitterion (V)

Fig. 1. Schematic drawings of ε-aminocaproic acid and 3-hydroxy-5-(3-aminopropyl)isoxazole.

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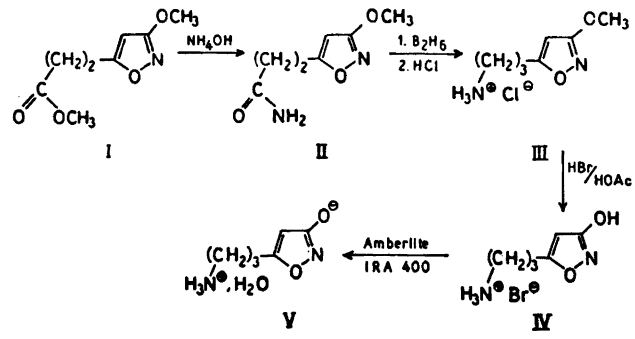

Scheme 1.

RESULTS AND DISCUSSION

The synthesis of 3-hydroxy-5-(3-aminopropyl)isoxazole zwitterion (V) is based on methyl 3-(3-methoxyisoxazol-5-yl)propionate (I), which is converted into 3-(3-methoxyisoxazol-5-yl)-propionamide (II). Subsequent reduction of (II) with diborane gives the corresponding primary amine, isolated from the reaction mixture as its hydrochloride (III). (III) is cleaved by hydrogen bromide in glacial acetic acid to 3-(3-hydroxyisoxazol-5-yl)propylammonium bromide (IV), from which the isoxazole enol-betaine (V) is isolated using a strongly basic ion exchange resin.

The structure determinations of (II – V) are based on IR, UV, and ^{1}H NMR spectroscopy and supported by elemental analyses. The spectroscopic data arising from the 3-methoxy- and 3-hydroxyisoxazole moieties of (II, III) and (IV), respectively, are in accordance with the general findings described by Jacquier *et al.*[7] The aromatic character of the isoxazole nucleus of (V) is evident from IR absorption bands at 1610 and 1520 – 1450 cm^{-1},[8] and absorptions over the range 3600 – 2000 and at 2170 cm^{-1} suggest ammonium salt character of (V). The UV maximum of (V) at 211 nm is in agreement with that observed (210 nm) for homomuscimol.[8] Some spectroscopic data of the compounds (II – V), which are all new, and the pK_A values of (V) are listed in Table 1.

The molecular structure of (V) is unambiguously confirmed by the results of the X-ray diffraction analysis. The dimensions and the conformation of the molecule are shown in Figs. 2a and 2b.

The isoxazole ring is planar within the limits of experimental error; Table 2 lists the displacements of some atoms from the least-squares plane through this ring. The least-squares plane through the planar *trans-zigzag* aminopropyl side chain makes an angle of 11.3° with the isoxazole ring plane (Table 2). The three hydrogen atoms are tetrahedrally arranged about the nitrogen atom. Their positions correspond to a 5 – 15° rotation about the C – N bond relative to that for a strictly staggered conformation.

Tables 3 and 4 and Figs. 2a and 2b show some

Table 1. IR and UV data of (II – V) and the pK_A values of (V).

	IR data [a] (cm^{-1})	UV data [b] λ_{max}(nm)	$\varepsilon \times 10^{-3}$	pK_A values [c]
II	3370(s), 3200(s), 1660(s), 1635(s), 1615(s), 1515(s), 1460(s)	211	6.86	
III	3700 – 2400(s), 2040(w), 1615(s), 1515(s), 1505(s), 1460(s)	211	5.58	
IV	3700 – 2100(s), 2000(w), 1620(s), 1525(s), 1465(s)	209	6.02	
V	3600 – 2000(s), 2170(m), 1710(w), 1610(s), 1520 – 1450(s)	211	6.16	5.37 ± 0.04 10.36 ± 0.04

[a] The IR spectra were recorded in the solid state (KBr). [b] The UV spectra were recorded in ethanol solutions. [c] The pK_A values were determined by titrations in aqueous solutions at 17 °C.

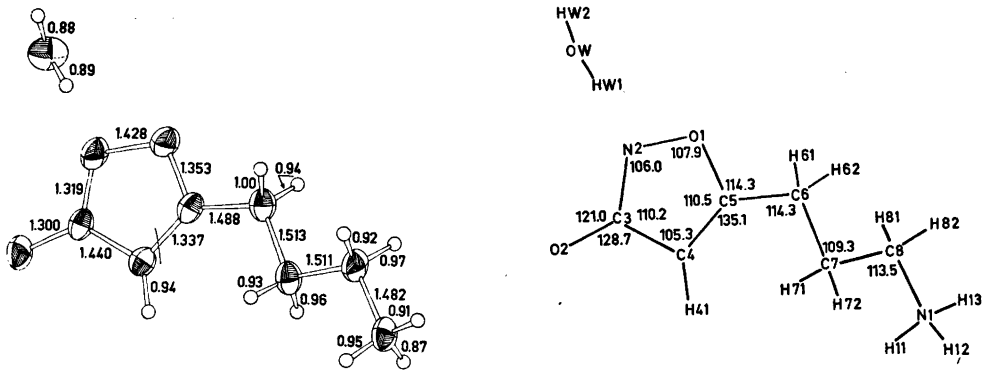

Fig. 2. (a) Bond lengths (Å), and thermal ellipsoids for the non-hydrogen atoms drawn to enclose 50 % probability. (b) The numbering of the atoms, and some bond angles (°).

Table 2. Distances of atoms from least squares planes (Å). The equations of the planes are in direct (unit-cell) space.

Atom	Deviation	Atom	Deviation
I. Isoxazole ring			
Equation:	$-10.1648x+0.7429y+13.3311z-0.2225=0$		
O(1)	−0.001	O(2)[a]	0.053
N(2)	−0.004	C(6)[a]	0.063
C(3)	0.006	C(7)[a]	0.176
C(4)	−0.007	C(8)[a]	0.471
C(5)	0.005	N(1)[a]	0.601
II. 3-Aminopropyl side chain			
Equation:	$14.1011x-0.6273y-13.3061z-1.9908=0$		
N(1)	−0.0003	C(6)	−0.0003
C(8)	0.0003	C(5)[a]	−0.2336
C(7)	0.0003		

[a] These atoms were not included in the calculation of the least squares plane.

*Table 3. Intra*molecular distances (Å). The estimated standard deviations ($\times 10^2$ for bonds to hydrogen, $\times 10^3$ for others) of the distances are given in parentheses.

O(1)−N(2)	1.428(2)	C(7)−H(71)	0.93(3)
N(2)−C(3)	1.319(3)	C(7)−H(72)	0.96(2)
C(3)−C(4)	1.440(3)	C(8)−H(81)	0.92(2)
C(4)−C(5)	1.337(3)	C(8)−H(82)	0.97(3)
C(5)−O(1)	1.353(2)	N(1)−H(11)	0.95(3)
C(5)−C(6)	1.488(3)	N(1)−H(12)	0.87(3)
C(6)−C(7)	1.513(3)	N(1)−H(13)	0.91(2)
C(7)−C(8)	1.511(3)	O(W)−H(W1)	0.89(2)
C(8)−N(1)	1.482(3)	O(W)−H(W2)	0.88(3)
C(3)−O(2)	1.300(2)	N(1)···N(2)	7.252(4)
C(4)−H(41)	0.94(2)	N(1)···O(1)	6.165(3)
C(6)−H(61)	1.00(2)	N(1)···O(2)	7.925(5)
C(6)−H(62)	0.94(3)		

Table 4. Valency angles (°). The estimated standard deviations (× 10 for angles not involving hydrogen) of the angles are given in parentheses.

C(5) – O(1) – N(2)	107.9(1)	H(61) – C(6) – H(62)	103(2)
O(1) – N(2) – C(3)	106.0(1)	C(6) – C(7) – H(71)	109(1)
N(2) – C(3) – C(4)	110.2(2)	C(6) – C(7) – H(72)	110(1)
N(2) – C(3) – O(2)	121.0(2)	C(8) – C(7) – H(71)	108(1)
C(4) – C(3) – O(2)	128.7(2)	C(8) – C(7) – H(72)	113(1)
C(3) – C(4) – C(5)	105.3(2)	H(71) – C(7) – H(72)	108(2)
C(4) – C(5) – O(1)	110.5(1)	C(7) – C(8) – H(82)	110(1)
C(6) – C(5) – O(1)	114.3(2)	C(7) – C(8) – H(81)	110(1)
C(6) – C(5) – C(4)	135.1(2)	N(1) – C(8) – H(82)	107(1)
C(5) – C(6) – C(7)	114.3(2)	N(1) – C(8) – H(81)	108(1)
C(6) – C(7) – C(8)	109.3(2)	H(82) – C(8) – H(81)	107(2)
C(7) – C(8) – N(1)	113.5(2)	C(8) – N(1) – H(11)	110(1)
C(3) – C(4) – H(41)	129(1)	C(8) – N(1) – H(13)	107(1)
C(5) – C(4) – H(41)	126(1)	C(8) – N(1) – H(12)	110(1)
C(5) – C(6) – H(61)	110(1)	H(11) – N(1) – H(13)	109(2)
C(5) – C(6) – H(62)	109(1)	H(11) – N(1) – H(12)	110(2)
C(7) – C(6) – H(61)	108(1)	H(13) – N(1) – H(12)	111(2)
C(7) – C(6) – H(62)	111(1)	H(W1) – O(N) – H(W2)	102(2)

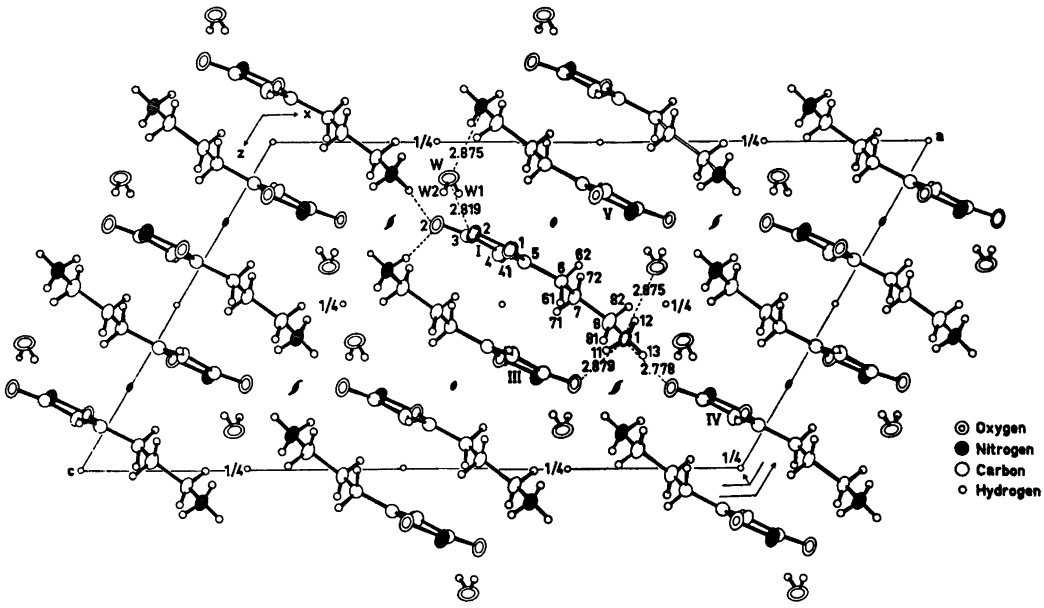

Fig. 3. Projection of the structure down the *b* axis. Some of the shorter *inter*molecular contacts are indicated.

calculated *intra*molecular distances and angles. The bond lengths and angles appear consistent with the general pattern of 3-hydroxy-5-(ω-aminoalkyl)isoxazole zwitterions.[2,3] The *intra*molecular distance $N(1)^+ \cdots O(2)^-$ is 7.925 Å.

The anisotropic thermal parameters of the non-hydrogen atoms are given in Table 6 and are drawn schematically in Fig. 2a.

The packing of the molecules in the crystal structure is determined by a system of hydrogen bonds. All hydrogen atoms that are covalently bonded to nitrogen or oxygen atoms are utilized

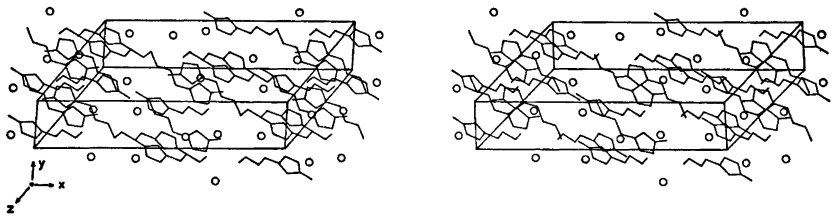

Fig. 4. Stereo diagram illustrating the molecular packing.

in the formation of hydrogen bonds. The zwitterions are inclined at about 13° to the ($\bar{1}01$) planes and, since they are approximately planar, form layers as shown in Figs. 3 and 4. Those zwitterions related by the *n*-glide plane are bound head to tail by the hydrogen bond $N(1)-H(13)\cdots O(2)_{IV}$, thereby leading to infinite chains in the [101] direction. These chains are interlinked on one side by double hydrogen bonds [$N(1)-H(11)\cdots O(2)_{III}$; $O(2)\cdots H(11)_{III}-N(1)_{III}$], formed between pairs of molecules related by a centre of symmetry. The spaces between these pairs of layers are filled with water molecules, each of which is linked by hydrogen bonds in directions almost parallel to the *b* [$O(W)-H(W1)\cdots N(2)_I$; $O(W)-H(W2)\cdots O(2)_{II}$] and *c* [$O(W)\cdots H(12)_V-N(1)_V$] axes to three different 3-hydroxy-5-(3-aminopropyl)-isoxazole molecules. In addition the $N(1)\cdots O(W)_{IV}$ distance is short, presumably because of the strong attractive interactions between the molecules. All other *inter*molecular contacts are greater than 3.2 Å. A summary of the geometry of the close contacts is shown in Table 5.

EXPERIMENTAL

The determination of melting points, the recording of IR, UV, and ^1H NMR spectra, and the performance of microanalyses were accomplished as described in a previous paper.[9]

The pH values were measured on a Radiometer pH meter 26, and the pK_A values were determined according to the method of Albert and Serjeant [10] as described previously.[3]

The computations were performed on a GIER computer and an IBM 360/75 computer using *INDIFF*,[11] a local version of *The N. R. C. 2A Picker Data Reduction Program*,[12] *The X-Ray System*,[13] and *ORTEP*.[14] The X-ray atomic scattering factors used were those of Cromer and Mann [15] for oxygen, nitrogen, and carbon, and of Stewart, Davidson, and Simpson [16] for hydrogen.

Synthesis

3-(3-Methoxyisoxazol-5-yl)propionamide (II). A mixture of 3.20 g (17 mmol) of methyl 3-(3-methoxyisoxazol-5-yl)propionate (I) [17] and aqueous ammonia (170 ml; ϱ 0.88) was stirred until a clear solution was obtained. This was allowed to stand at 25 °C for 18 h. Evaporation *in vacuo* afforded 3.0 g of crystalline product, which was recrystallized (acetone) to give 2.21 g (75 %) of (II) as colourless crystals, m.p. 142.0–143.0 °C. (Found: C 49.60; H 6.03; N 16.42. Calc. for $C_7H_{10}N_2O_3$: C 49.40; H 5.92; N 16.46). ^1H NMR data [$CDCl_3$-DMSO-d_6 (1:1)]: δ 7.5–7.1 and 6.8–6.5 (two broad signals, total 2 H, $CONH_2$); 5.75 (s, 1 H, $C=CH-C$); 3.84 (s, 3 H, $O-CH_3$); 3.1–2.7 (m, 2 H, $CH_2-CH_2-C=$); 2.6–2.2 (m, 2 H, $CO-CH_2-CH_2$).

3-(3-Methoxyisoxazol-5-yl)propylammonium chloride (III). To 100 ml of ice-cooled tetra-

Table 5. Close *inter*molecular contacts involving hydrogen and non-hydrogen atoms.

C–A–H	B	B equipoint	A–H Å	A···B Å	H···B Å	∠AHB deg.	∠CAB deg.
$O(W)-H(W1)\cdots N(2)_I$		(x,y,z)	0.89(2)	2.819(2)	1.94(2)	173(3)	
$O(W)-H(W2)\cdots O(2)_{II}$		$(x,y+1,z)$	0.88(3)	2.737(2)	1.86(3)	174(3)	
$C(8)-N(1)-H(11)\cdots O(2)_{III}$		$(1-x,-y,1-z)$	0.95(3)	2.879(3)	1.95(3)	168(1)	115.5(1)
$C(8)-N(1)-H(12)\cdots O(W)_V$		$(1-x,y-1,\frac{1}{2}-z)$	0.87(3)	2.875(3)	2.07(3)	153(2)	91.6(1)
$C(8)-N(1)-H(13)\cdots O(2)_{IV}$		$(\frac{1}{2}+x,\frac{1}{2}-y,\frac{1}{2}+z)$	0.91(2)	2.778(2)	1.90(2)	161(2)	94.0(1)
$C(8)-N(1)-H(12)\cdots O(W)_{IV}$		$(\frac{1}{2}+x,\frac{1}{2}-y,\frac{1}{2}+z)$	0.87(3)	3.073(3)	2.57(2)	118(1)	152.1(2)

hydrofuran containing diborane, externally generated[18] from 4.75 g (125 mmol) of sodium borohydride in diglyme (125 ml) and 25.5 g (180 mmol) of boron trifluoride etherate in diglyme (50 ml), was added during a period of 15 min a solution of 5.95 g (35 mmol) of (II) in tetrahydrofuran (125 ml). The mixture was refluxed for 19 h, and after cooling to 25 °C followed by careful addition of 6 M hydrochloric acid (50 ml) the solution was evaporated to dryness *in vacuo*. Upon addition of water (10 ml) and a 50 % aqueous solution of potassium hydroxide (40 ml) the mixture was extracted with two 50 ml portions of ether. The combined ether phases were dried and evaporated *in vacuo* to give 3.3 g of an oil. The oily product was dissolved in ethanol (13 ml) and upon addition of an ethanolic solution of hydrogen chloride, prepared from ethanol (100 ml) and acetyl chloride (12 ml), followed by addition of ether (18 ml) (III) was allowed to crystallize at 5 °C for 18 h to give 2.88 g (43 %) as colourless crystals, m.p. 129 – 131 °C (decomp.). (Found: C 43.40; H 6.80; N 14.65; Cl 18.43. Calc. for $C_7H_{13}ClN_2O_2$: C 43.65; H 6.80; N 14.54; Cl 18.41). ^{1}H NMR data [DMSO-d_6-$CDCl_3$ (3:2)]: δ 8.7 – 8.0 (broad, 3 H, NH_3^+); 5.93 (s, 1 H, C=C*H*–C); 3.87 (s, 3 H, O–CH_3); 3.1 – 2.5 (m, 4 H, NH_3^+–CH_2–CH_2–CH_2–C=); 2.3 – 1.7 (m, 2 H, CH_2–CH_2–CH_2).

3-(3-Hydroxyisoxazol-5-yl)propylammonium bromide (IV). 1.40 g (7.3 mmol) of (III) were dissolved in 11 ml of glacial acetic acid containing 43 % of hydrogen bromide. The solution was heated in an oil bath for 30 min, during which time the temperature rose to 90 °C. A further amount of 11 ml of the above mentioned reagent was carefully added, and the solution was refluxed (bath temperature: 90 – 100 °C) for 40 min. After cooling to 25 °C the reaction mixture was evaporated to dryness *in vacuo*. Recrystallization (methanol-ether) gave 1.10 g (68 %) of (IV) as colourless crystals, m.p. 193 °C (decomp.). (Found: C 32.30; H 5.02; N 12.60; Br 35.74. Calc. for $C_6H_{11}BrN_2O_2$: C 32.30; H 4.97; N 12.55; Br 35.82). ^{1}H NMR data (DMSO-d_6): δ 11.5 – 10.8 (broad, 1 H, O*H*); 8.5 – 7.4 (broad, 3 H, NH_3^+); 5.82 (s, 1 H, C=C*H*–C); 3.0 – 2.4 (m, 4 H, NH_3^+–CH_2–CH_2–CH_2–C=); 2.2 – 1.5 (m, 2 H, CH_2–CH_2–CH_2).

3-Hydroxy-5-(3-aminopropyl)isoxazole zwitterion (V). A solution of 900 mg (4.0 mmol) of (IV) in water (10 ml) was passed through a column containing an ion exchange resin [Amberlite IRA 400, (OH), 90 ml] using acetic acid (1 M) as an eluent. The fractions containing (V) were collected and evaporated *in vacuo* to give an oil. The oily residue was dissolved in water (15 ml), and upon addition of ethanol (35 ml) (V) was allowed to crystallize at 5 °C for 18 h to give 280 mg (43 %) as colourless crystals, m.p. 136 – 137 °C (decomp.). (Found: C 44.80; H 7.58; N 17.39. Calc. for $C_6H_{10}N_2O_2.H_2O$: C 44.99; H 7.55; N 17.49). [Found after drying of (V) over P_2O_5 (16 h; 75 °C; 0.05 mmHg): C 50.55; H 6.96; N 19.69. Calc. for $C_6H_{10}N_2O_2$: C 50.69; H 7.09; N 19.71]. ^{1}H NMR data [D_2O (sodium 3-(trimethylsilyl)propanesulfonate was used as an internal standard)]: δ 5.52 (s, 1 H, C=C*H*–C); 4.69 (s, 5 H, DO*H*); 3.2 – 2.8 and 2.8 – 2.4 (two t, total 4 H, NH_3^+–CH_2–CH_2–CH_2–C=); 2.2 – 1.5 (quintet, 2 H, CH_2–CH_2–CH_2).

X-Ray analysis

Colourless prismatic crystals of 3-hydroxy-5-(3-aminopropyl)isoxazole monohydrate were grown at room temperature by diffusion of absolute ethanol into an aqueous solution of the compound.

Crystal data. 3-Hydroxy-5-(3-aminopropyl)-isoxazole monohydrate, $C_6H_{10}N_2O_2.H_2O$, M = 160.18. Monoclinic, $a = 23.45(1)$, $b = 5.768(1)$, $c = 13.483(4)$ Å, $\beta = 120.33(3)°$, $U = 1574.0$ Å^3, $D_m = 1.35$ g cm^{-3}, $Z = 8$, $D_c = 1.352$ g cm^{-3}. Linear absorption coefficient for X-rays [λ (Mo$K\alpha$) = 0.7107 Å], $\mu = 1.17$ cm^{-1}. Number of electrons per unit cell, $F(000) = 688$. Space group $C2/c$ from systematically absent reflections: hkl when $h+k$ odd, $h0l$ when l odd, and from intensity statistics.

The unit-cell parameters were refined by least-squares techniques from the diffractometer-measured θ angles observed for 40 reflections well distributed in reciprocal space. The crystal density was measured by flotation in a chlorobenzene/bromobenzene mixture.

Data collection. Three-dimensional diffraction data were measured at room temperature on a Nonius three-circle automatic diffractometer using graphite monochromated Mo$K\alpha$ radiation. The ω scan technique with a scan speed of 1.2° min^{-1} was employed. Background counts were taken for half the scanning time at each of the scan range limits. One standard reflection was measured after every 25 reflections.

All the data were measured from a single crystal with approximate dimensions 0.25 × 0.32 × 0.30 mm. The crystal was mounted with [010] along the ϕ axis of the goniostat.

Out of the 1725 independent reflections measured in the range $2.5° \leq \theta \leq 27°$, 1231 had $I_{net} \geq 3.0\sigma(I)$, where $\sigma(I)$ is the standard deviation from counting statistics. These were regarded as observed reflections, whereas the remaining reflections were regarded as unobserved and excluded from the refinement procedure. Lorentz and polarization corrections were applied, but no absorption corrections were made owing to the low absorption coefficient.

Structure determination. The observed structure amplitudes were brought to an absolute scale by Wilson statistics and normalized structure amplitudes, $|E(hkl)|$, were calculated.

The phase problem was solved by direct methods using the programs of the X-Ray 72 System.[18] Of the highest thirteen peaks in an E-map based on 92 $E(hkl)$'s with $|E(hkl)| \geq 1.5$, eleven corresponded to all the "heavy" (nitro-

gen, oxygen, and carbon) atoms in the asymmetric unit. Individual atomic parameters of this model were refined, first with isotropic and then anisotropic thermal parameters using the full-matrix least-squares method. On convergence the R value was 0.093. The quantity minimized was $\sum w(|F_o|-|F_c|)^2$, where weights were initially taken as unity.

After having omitted from the data set the strongest observation, *i.e.* $\bar{4}04$, which was considered to be severely affected by extinction, a three-dimensional difference synthesis was computed. The subsequent map showed maxima with peak heights of $0.4-0.7$ e Å^{-3} in positions expected for all the hydrogen atoms, and there were no extraneous peaks greater than the lowest hydrogen atom. Introduction of the hydrogen atoms in the refinement, with the isotropic temperature factors of the connected non-hydrogen atoms as constant parameters reduced the R value to 0.050 for 1231 independent observations. Among the strongest reflections twelve were considered to be affected by extinction, and were omitted from the data set.

The least-squares refinement was completed with the introduction of a weighting scheme of the form: $w=1/(1+[(|F_o|-b)/a]^2)$, where $a=17.50$ e and $b=18.75$ e. On the last cycle of least-squares refinement shifts of all parameters were less than one tenth of their estimated standard deviations and the final R value is 0.039 for 1218 independent observations. Tables 6 and 7 list the final positional and thermal parameters for the non-hydrogen and hydrogen atoms, respectively. From these parameters the terminal set of structure factors, listed with the observed data in Table 8, was computed. Comparison of the 92 signs determined by direct methods with the corresponding phases computed from the refined structure shows that all had been correctly assigned.

Table 6. Final positional and thermal ($\times 10^4$ Å^2) parameters for non-hydrogen atoms. The estimated standard deviations of positional and thermal parameters ($\times 10^4$) are given in parentheses. The temperature expression is of the form:
$\exp[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2hka^*b^*U_{12}+2hla^*c^*U_{13}+2klb^*c^*U_{23})]$

	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.4590(1)	0.6042(2)	0.3329(1)	250(6)	298(7)	407(7)	35(5)	121(5)	12(6)
N(2)	0.3897(1)	0.5642(3)	0.2821(1)	209(7)	365(9)	372(8)	64(6)	100(6)	21(7)
C(3)	0.3829(1)	0.3391(3)	0.2902(1)	214(8)	353(9)	231(8)	50(7)	94(6)	14(7)
C(4)	0.4464(1)	0.2268(3)	0.3439(2)	225(8)	291(9)	324(9)	44(7)	120(7)	31(8)
C(5)	0.4902(1)	0.3975(3)	0.3687(1)	230(8)	326(9)	226(7)	60(7)	105(6)	16(7)
C(6)	0.5635(1)	0.4121(4)	0.4282(1)	223(8)	380(10)	258(8)	−1(8)	107(7)	6(8)
C(7)	0.5986(1)	0.1838(4)	0.4761(2)	208(8)	385(11)	325(9)	2(8)	108(7)	8(8)
C(8)	0.6718(1)	0.2276(3)	0.5516(2)	198(8)	365(10)	353(9)	7(8)	102(7)	−26(8)
N(1)	0.7108(1)	0.0137(3)	0.6030(1)	199(7)	399(9)	326(8)	1(6)	104(6)	−16(7)
O(2)	0.3250(1)	0.2480(2)	0.2547(1)	193(6)	434(8)	395(7)	22(6)	113(5)	21(6)
O(10)	0.3025(1)	0.8774(3)	0.1127(1)	447(8)	381(8)	324(7)	19(7)	90(6)	29(6)

Table 7. Final positional and thermal ($\times 10^3$ Å^2) parameters for hydrogen atoms. The estimated standard deviations ($\times 10^3$) of the coordinates are given in parentheses.

	x/a	y/b	z/c	U_{iso}
H(41)	0.456(1)	0.068(4)	0.359(2)	28
H(61)	0.580(1)	0.525(4)	0.493(2)	29
H(62)	0.576(1)	0.479(4)	0.379(2)	29
H(71)	0.583(1)	0.120(3)	0.522(2)	31
H(72)	0.588(1)	0.078(4)	0.414(2)	31
H(81)	0.679(1)	0.327(4)	0.610(2)	31
H(82)	0.689(1)	0.300(4)	0.507(2)	31
H(11)	0.693(1)	−0.072(4)	0.641(2)	31
H(12)	0.711(1)	0.071(4)	0.550(2)	31
H(13)	0.752(1)	−0.060(4)	0.656(2)	31
H(W1)	0.331(1)	0.776(4)	0.162(2)	38
H(W2)	0.307(1)	0.996(4)	0.158(2)	38

Table 8. Observed structure amplitudes $|F_o|$ and calculated structure factors F_c (in electrons × 10). Within each group of reflections with constant k and l, the column list, from left to right, h, $10|F_o|$, and $10F_c$. Reflections considered unobserved or affected by extinction are marked with one or two asterisks, respectively.

	H,0,0		-26	67	68	-11	465	457	-9	570	583		H,1,14				H,2,4					
						-13	796	774	11	186	-188											
4	366	-342		H,0,10		15	335	339	-11	83	79	1	155	146	0	660	-667					
6	221	211				-15	45	27	13	79	-97	-1	124	120	2	454	462					
8**	1128	-1194	0	82	-81	-19	285	-289	15	129	129	-7	108	-106	-2	364	353					
10	655	648	2	228	-232	23	64	-55	-15	289	291	-9	159	158	-4	64	-77					
12	350	-337	-2	139	-128	-23	67	64	17	81	-77	-17	57	-51	6	352	-345					
14	551	-541	4	99	-98	-25	117	-120	-17	309	-309	-21	200	-199	-6	294	-278					
16	192	195	-4	85	-86	-27	129	128	-23	398	-401	-23	122	-122	8	334	349					
18	476	494	-6	481	-481				-25	90	-91	-25	78	-82	10	319	-324					
20	88	96	8	168	169		H,1,3		-29	79	-88				-10	105	-90					
22	56	-53	-8	259	252								H,1,15		12	57	-64					
24	95	95	10	188	191	1	663	-703		H,1,8					-12	258	-252					
			-10	520	527	-1	623	677				-1	108	95	14	109	115					
			12	170	-173	3	726	762	1	98	-92	-3	72	-75	-16	235	216					
	H,0,2		-12	385	-378	-3**	1321	1709	-1	170	-180	-5	76	-78	18	70	60					
			-14	289	302	5	514	-534	3	210	198	-11	155	-160	-18	179	-177					
			-18	226	227	-5**	940	-1053	-3	101	90	-13	136	-141	20	80	79					
0	499	-476	-20	493	493	7	131	-139	5	205	-204	-17	54	62	-20	127	128					
2	733	-789	-22	263	-263	-7	37	-40	-5	295	279	-19	101	106	-24	74	-72					
-2	427	454	-26	121	119	9	530	537	7	60	54				-26	201	199					
4	87	-83	-28	76	72	-9	69	56	-7	699	716		H,1,16									
-4	706	712				11	268	-264	-9	161	161						H,2,5					
6	739	-735		H,0,12		-11	404	-392	11	131	137	-7	93	-107								
-6	105	119				13	85	87	-11	148	148	-11	91	108	0	64	62					
8	737	-727	0	56	-61	-13	380	387	13	164	178	-13	181	181	2	451	455					
-8	221	202	2	223	213	15	188	190	-13	113	103	-15	153	152	4	166	-160					
10	376	-366	-2	117	-121	-15	78	66	15	154	-154	-19	82	90	-4	259	265					
-10	919	967	4	82	76	17	146	-141	-15	176	172				6	99	-85					
12	181	184	-4	135	125	-17	170	-170	19	195	-193		H,1,17		-6	141	-130					
-12	457	439	6	90	-96	-19	340	359	-21	49	-41				8	437	-449					
14	411	403	-6	183	-185	-21	337	347	-23	100	94	-15	103	-110	-8	112	-103					
-14	583	581	8	387	-396	-25	105	86	-25	144	155				10	470	-482					
16	107	103	-10	363	-367				-27	94	-91		H,2,0		-10	439	-427					
-16	570	-585	-12	316	-307		H,1,4								12	107	-113					
18	100	-91	-14	51	-62					H,1,9		0	213	-182	-12	396	-394					
-18	382	-372	-16	143	143	1	387	409				2	160	146	14	162	-166					
20	68	-68	-24	67	-68	-1	517	-537	1	356	358	4	537	535	-14	115	106					
-20	326	325	-26	131	-172	3	311	-317	-1	64	48	6	669	-599	16	71	59					
22	128	128				-3**	1269	-1533	-5	239	237	8	369	360	-16	90	-87					
-22	142	-144		H,0,14		5	362	371	7	164	164	10	281	280	18	82	70					
-24	120	115				-5	176	-159	-7	254	-239	12	477	-460	-20	164	-161					
			2	179	184	7	265	-264	11	147	150	14	457	457	-24	166	164					
	H,0,4		-2	204	200	-7	621	-662	-11	161	151	16	93	-103	-26	80	80					
0	476	-516	-4	80	-63	9	286	282	13	63	65	18	148	-152	-28	109	93					
2	672	688	8	219	217	-9	284	295	-13	101	-112	20	139	147								
-2**	1063	-1232	-10	67	-64	11	94	99	-15	84	-88	22	209	-196			H,2,6					
4	62	-49	-12	125	-123	-11	311	-302	-21	252	247											
-4**	1761	-3298	-14	180	-174	13	330	-334	-23	263	265		H,2,1		0	78	-69					
6	77	-73	-16	149	164	-13	160	-155	-25	184	-183				2	89	-99					
-6	273	-278	-20	102	-94	15	358	346	-27	93	-89	0	117	111	-2	215	-221					
8	303	299	-22	164	-174	17	137	-149				2	573	-583	4	325	-326					
-8	588	622	-24	130	-119	-17	89	75		H,1,10		-2	203	-206	-4	343	330					
10	230	235	-26	103	110	19	181	175				4	162	151	6	455	467					
-10	255	-255		H,0,16		-21	230	-230	1	186	-182	6	392	-381	-6	250	250					
12	424	425				-23	54	24	-1	119	111	-6	961	947	8	62	66					
-12	561	581	-6	66	69	-25	85	77	3	129	-124	8	50	-26	-8	222	-227					
14	674	-715	8	86	-83	-27	62	40	-3	122	115	-8	259	260	10	67	-88					
-14	259	-256	-10	84	93		H,1,5		-5	171	-169	10	424	403	-10	44	-41					
16	246	-249	-12	276	275				7	186	184	-10	288	271	12	164	159					
-16	293	278	-14	50	46	-1	277	281	9	53	49	12	292	290	-12	415	404					
18	87	102	-16	173	175	3	246	-249	-9	151	-153	14	600	619	14	141	-135					
-18	314	307	-20	96	-111	-3	443	-444	-11	189	191	-14	109	-96	-14	363	-367					
20	136	-139	-22	127	123	5	66	-79	-15	188	-188	16	211	218	16	85	52					
-20	410	-413				-5	150	158	-17	315	318	-16	238	234	-18	182	-188					
-22	199	-215		H,1,0		7	171	-163	-19	148	149	-20	118	-124	-20	357	-355					
						-7	65	-70	-21	272	272	-22	115	-117	-24	242	244					
	H,0,6		1**	984	1052	9	65	73	-25	62	-66	-24	125	-117	-28	107	115					
			3**	1101	1193	-9	256	-257	-29	81	-84		121	-127								
0	201	-195	5**	955	-1010	11	83	-87					H,2,2				H,2,7					
2	296	293	7	672	683	-11	483	485		H,1,11					0	465	-463					
-2**	1167	1353	9	127	-132	13	205	-213				0	155	138	-2	94	-82					
4	570	563	11	186	-181	-13	78	-69	-3	141	140	-2	292	-280	4	95	-82					
-4	282	-277	13	94	-71	15	151	-146	5	214	-217	4	415	396	-4	434	-438					
-6	784	-810	15	166	-162	-15	96	-89	-5	100	-97	-4	117	-116	6	64	-70					
8	213	219	17	401	403	-17	117	-115	7	84	93	6	138	137	8	60	52					
-8	223	206	19	40	3	19	75	79	-7	253	257	-6	291	282	-8	357	367					
10	179	-181	23	96	-92	-19	399	-402	9	114	110	-8	763	-716	10	131	-132					
-10	580	-588	25	66	65	-21	55	63	-9	351	347	10	473	-473	-10	79	-81					
12	323	-320				-23	135	141	-13	80	-81	-10	284	283	12	56	65					
-12	133	126		H,1,1		-27	85	89	-15	187	-186	12	107	-105	-12	274	284					
14	71	59							-17	75	-61	-12	195	183	14	114	110					
-14	470	-483	1	195	186		H,1,6		-23	68	76	14	61	46	-14	171	-170					
16	97	98	-1**	768	894				-25	137	137	-14	215	212	16	101	94					
-16	568	-563	3	546	-559	1	64	-62	-27	148	147	-16	335	338	-16	79	-71					
18	139	-141	-3	412	437	-1	232	235				18	174	178	-20	401	-414					
-20	345	350	-5	221	-224	3	210	-206		H,1,12		-18	193	-191	-22	279	-273					
-22	53	41	7	238	249	-3	253	265				20	123	-122	-24	111	-116					
-24	282	-274	-7	40	56	5	429	430	-3	81	67	-20	98	-106	-26	74	70					
-26	125	130	9	62	-62	-5	448	448	-5	103	106	-26	69	-65	-28	63	70					
-28	85	-82	-9	378	-346	7	266	273	7	148	-149											
			11	347	344	-7	691	-707	-7	102	-94		H,2,3				H,2,8					
	H,0,8		-13	453	445	11	215	-217	-11	267	-271	0	635	634	-2	101	-86					
			15	53	55	-11	97	79	-15	120	-113	2	444	432	-4	172	169					
0	268	280	17	496	519	13	58	-61	-17	48	-51	-2	527	-551	6	149	142					
2	98	-103	-17	162	164	-13	218	-203	-19	108	-115	4	411	410	-6	202	-198					
-2	447	-455	-21	195	-183	15	57	59	-21	56	55	6	450	449	8	65	53					
-4	443	460	-23	60	54	-15	227	-220	-23	116	120	-6	291	-306	-8	223	229					
6	222	-225	-25	87	-93	-17	619	-618	-25	64	-67	8	219	-221	10	77	79					
-6**	1068	1152				-19	57	60				-8	375	-366	12	91	-83					
8	362	-367		H,1,2		-23	68	68		H,1,13		16	90	88	-14	311	314					
-8	590	576				-25	80	73				-10	261	271	-18	74	60					
10	434	453	1	614	-620	-27	60	-60	3	103	-94	12	82	81	-20	140	-143					
-10	288	297	-1	538	-592	-29	92	99	-3	182	-178	-12	129	-144	-22	53	-62					
12	243	249	3	48	-33				-7	53	-60	-14	313	295	-24	60	61					
-12	194	-194	-3**	718	804		H,1,7		-9	154	-143	-16	414	414								
14	107	109	5	398	390				-11	286	281	18	211	-208			H,2,9					
-14	109	-117	-5	75	66	-1	130	-140	-15	150	-148	-18	367	376								
-16	101	103	7	214	-209	3	53	62	-17	152	150	20	66	-94	0	89	-93					
-18	82	-96	-7	149	151	-5	85	-83	-19	62	-52	-20	280	285	2	48	49					
-20	171	-178	9	617	-609	-5	515	-498	-21	59	67	-22	98	-96	-2	48	-43					
-22	166	173	-9	338	-329	-7	649	-665	-25	279	-279	-24	61	-58	4	422	427					
-24	140	133	11	338	-331	9	202	204														

Table 8. Continued.

	H,2,9		5	500	-489	5	159	166	-19	82	-79		H,4,6		3	178	-185
			-5	407	375	-5	298	305							7	95	87
-4	191	-199	7	269	250	7	125	-130	10	H,4,0		0	151	-155	9	70	62
6	217	209	-7	180	145	-7	143	145				2	125	125	11	42	51
-6	127	123	9	77	66	9	116	-113	0	77	-91	4	93	-98	15	90	83
8	183	178	-9	373	361	-9	67	80	2	389	-392	6	86	-96	17	81	-73
-8	79	-70	11	234	237	11	246	252	4	151	-145	-6	279	-280			
10	114	102	-11	89	-98	-11	152	159	6	122	132	8	168	-172		H,5,1	
-10	86	-80	13	622	643	-13	85	-89	8	212	-208	-8	241	243			
-12	313	315	-13	1336	1336	-15	92	-91	10	115	-114	10	59	62	1	126	-117
-14	86	82	15	285	281	-19	246	-250	12	192	187	-10	151	-148	-1	130	136
-16	98	87	-15	117	115	-21	384	-378	16	52	-50	14	60	-55	3	99	98
-18	59	-57	-17	156	-164				18	137	147	-14	403	407	-3	66	-63
-22	111	114	-19	103	-101		H,3,8		20	143	-135	-16	102	104	5	295	298
			21	77	-64							-18	146	146	-5	174	169
	H,2,10		-23	89	-85	1	81	-89		H,4,1		-20	123	129	7	156	-152
						-1	218	213				-22	89	-86	9	223	-221
0	287	282		H,3,2		3	72	83	0	168	-154		H,4,7		-9	359	-350
2	214	-209				-3	187	-187	2	279	-262				-11	56	66
4	98	-98	1	673	688	-5	168	-165	-2	235	220				13	62	61
-4	118	109	-1	195	-200	7	96	-94	4	119	117	0	59	-56			
6	98	94	3	215	-202	9	57	66	-4	409	397	2	210	215		H,5,2	
-6	119	116	-3	60	65	-9	76	-69	6	100	99	-2	173	-171			
8	98	90	5	99	-106	-11	75	89	8	107	-112	-4	196	-192	1	196	200
-8	417	-411	-5	342	-336	-13	354	-358	-8	205	-217	6	158	-166	-1	194	-188
-12	84	89	7	123	114	-17	56	50	-10	117	-124	-6	70	-79	3	72	73
-14	78	-75	-7	48	-59	19	93	90	12	81	85	-8	76	68	-3	120	115
-18	157	161	-9	234	234	-21	101	99	-12	227	224	10	45	48	5	183	184
-22	175	178	11	409	408	-23	187	-179	14	206	205	-14	76	83	-5	83	-80
-24	125	131	-11	301	-281	-25	110	106	-14	240	241	-16	75	77	7	214	229
-26	262	-262	13	146	-140	-27	77	60	16	150	155	-18	180	-185	-7	240	-235
			-15	243	-238				-16	88	-98	-20	151	-152	-9	63	-54
	H,2,11		-19	82	74		H,3,9		-18	76	-88	-22	57	-43	11	87	93
			-23	111	99				-20	65	50	-24	77	-61	-11	244	-249
0	135	133				1	70	-63	-22	67	74				13	81	-84
2	62	-65		H,3,3		-1	68	-76					H,4,8		-19	57	31
-4	175	164				3	178	177		H,4,2							
-6	250	241	1	654	667	5	292	301								H,5,3	
8	82	-74	-1	228	231	-5	244	-237	2	104	-91	6	68	71			
-8	122	114	3	235	206	7	255	260	-2	264	263	-6	329	-343	1	143	143
-10	81	83	-3	527	-537	-7	68	-56	4	329	337	8	66	-76	3	89	91
-12	213	-211	5	161	160	9	189	187	-4	322	-318	-8	73	-66	-3	55	43
-16	72	64	-5	262	-245	-9	206	206	6	278	-271	10	55	-48	5	231	-233
-20	66	54	7	268	257	-13	280	263	-6	196	182	-10	210	-206	-5	102	-114
-24	235	231	9	92	-93	-15	104	95	8	348	356	-12	99	-106	7	234	-232
-26	224	229	-9	416	-410	-17	189	-183	-8	76	-68	-16	167	-176	-7	179	174
			11	145	144	-19	186	191	10	172	185	-18	153	158	9	113	110
	H,2,12		-11	349	348	-23	91	-93	-10	164	-164	-22	72	67	-9	222	223
			13	65	51	-25	89	94	-12	240	-244				11	55	65
0	52	-36	-13	107	95	-27	76	-70	14	131	138		H,4,9		-11	297	-290
2	145	-130	15	313	-321				-14	201	-208				13	55	-62
-2	129	132	-15	117	119		H,3,10		16	117	-114	0	119	118	-13	155	-155
6	127	-130	-17	727	732				18	61	46	-2	122	114	15	116	108
-6	99	92	-19	70	68	1	67	73	-18	64	-59	4	100	-98	-15	115	98
-10	98	-90	-21	63	-66	-1	172	-173	-20	113	116	6	127	128	-19	72	56
-12	81	-82	-23	60	65	3	121	128				-6	218	-222			
-16	96	-100	-25	170	-158	-7	64	71		H,4,3		8	164	166		H,5,4	
-18	119	-127				9	93	-88				-10	229	230			
-20	63	63		H,3,4		-9	73	69	0	345	346	-12	114	111	1	130	132
-22	81	-84				-17	98	-100	2	161	157	-14	159	-160	-1	94	103
-24	174	172	1	212	217	-19	108	-107	-2	109	115	-16	97	-102	3	65	-62
			-1	88	73	-23	111	-115	4	86	-105	-20	132	131	-3	451	464
	H,2,13		3	75	-62				-4	155	-145	-22	116	108	5	91	-102
			-3	179	173		H,3,11		6	209	-216	-24	81	-73	-5	372	372
0	181	-182	5	81	69				-6	133	-135				-7	77	68
2	192	-190	-5	74	-77	3	101	100	8	116	123		H,4,10		9	57	-57
-2	94	-86	7	59	-72	-3	106	106	-8	77	74				11	81	-77
4	150	-146	-9	409	412	-5	147	147	10	229	238	0	130	-126	-11	190	-188
-4	154	163	11	97	85	-7	265	262	12	66	-54	-2	82	81	13	91	87
-12	79	-77	-13	161	156	-9	105	-96	-12	157	-158	4	190	196			
-14	54	55	-15	278	-272	-11	79	-83	14	94	-94	6	122	-115		H,5,5	
-16	157	-149	17	103	-95	-15	54	-41	-14	128	128	-8	131	139			
-18	69	-55	-17	78	89	-17	106	103	-16	146	154	-14	130	127	1	56	-60
-20	85	84	-19	196	198	-21	64	71	18	107	109	-16	161	-175	-1	141	-149
			-21	117	-117	-23	203	207	-18	138	129	-18	228	-237	3	98	-106
	H,2,14		-25	116	-112	-25	139	138	-20	109	105	-20	87	-79	-3	100	94
												-22	124	-130	5	160	149
-2	68	74		H,3,5			H,3,12			H,4,4					7	148	163
-4	115	-112											H,4,11		-7	149	-140
-8	81	-77	-1	325	333	1	165	159	0	287	286				9	124	-126
-12	226	229	3	123	134	-1	129	-119	2	249	-271	-2	112	-115	-9	57	65
-14	54	-53	-3	243	236	-5	81	-79	-2	231	234	-4	117	115	-11	118	118
-24	109	-114	7	361	-370	-7	58	65	4	105	109	-6	139	133	-13	143	139
			-7	219	-212	-9	205	204	-4	54	41	-8	76	70	-15	89	-91
	H,2,15		9	315	-325	-11	120	-123	6	162	161	-10	58	67			
			-9	356	-334	-17	182	185	-6	536	557	-16	102	-94		H,5,6	
-10	201	-205	11	418	-423	-21	116	-119	8	126	-122	-18	76	-74			
-12	102	-109	-11	206	-191				-8	129	-126	-22	161	167	1	217	-215
-16	64	56	13	143	-141		H,3,13		-10	136	127				3	98	-122
-20	69	-95	15	108	119				12	58	64		H,4,12		-3	91	-89
			-15	51	-62	1	260	-258	-12	268	272				5	84	-79
	H,2,16		-17	74	-80	-3	92	89	14	73	-75	0	104	95	-5	111	101
			-21	83	76	-5	93	-76	-14	202	-204	-2	53	-40	11	40	-68
-10	113	-113				-7	130	123	16	116	108	-4	106	-102	-11	184	189
-12	66	44		H,3,6		-9	52	60	-18	79	-80	-6	99	104	-15	166	177
-14	137	132				-11	93	-94				-8	57	-64	-17	103	106
			1	102	-112	-13	63	-56		H,4,5		-10	245	250			
	H,3,0		-1	171	169	-15	101	-107				-12	103	102		H,5,7	
			3	197	201	-17	92	-104	0	69	-55	-16	235	237			
3	146	-132	-3	312	-313	-19	52	51	2	161	-153				1	69	71
5	254	-249	5	111	-118	-21	75	81	-2	299	295		H,4,13		-1	175	-176
7	86	-98	-5	64	-69	-23	73	-78	-4	67	65				3	312	315
9	170	-163	7	303	-310				6	55	47	-4	120	-114	5	57	-49
11	280	279	-7	156	158		H,3,14		-6	281	-276	-6	58	-45	-5	85	-82
13	41	41	9	136	142				-8	271	-276	-10	100	106	7	79	-77
15	94	-90	13	92	93	-5	65	56	10	205	-202	-14	120	-131	9	62	45
21	157	160	-15	175	164	-19	94	94	-10	147	150	-18	111	100	-13	240	-248
23	64	-56	-19	131	131	-23	57	58	12	178	-178				-15	223	222
			-27	107	-101				-12	184	189		H,4,14				
	H,3,1						H,3,15		-16	214	-214					H,5,8	
				H,3,7					-18	182	-186	-12	64	-58			
1	307	-294				-7	113	-106	-20	105	114				-1	79	91
-1	584	-590	-1	209	-199	-9	111	-104	-24	57	-59		H,5,0		3	50	41
3	53	-58	3	362	-355	-11	57	-54							-3	164	-168
-3	759	740	-3	634	-660	-15	84	65				1	421	-421	5	81	66

Table 8. Continued.

	H,5,8		-2	82	-81	21	30*	26		H,1,17			H,2,14			H,3,10		
			4	169	-180	-21	29*	14										
-5	104	-110	-10	92	103				-13	18*	-34	0	41*	32	-3	34*	46	
-7	276	-277	-12	98	95		H,1,3		-17	10*	12	-6	9*	-19	5	29*	7	
-9	347	-361	-14	112	-98							-10	9*	15	-6	13*	27	
-11	80	71	-16	98	93	19	9*	0		H,2,0		-16	37*	20	7	10*	-10	
-15	111	107				21	41*	10				-18	14*	-7	-11	20*	-37	
-19	88	-102		H,6,7		-23	46*	-50	24	8*	0	-20	34*	-41	-13	6*	-13	
						-27	11*	13				-22	52*	-43	-15	25*	-18	
	H,5,9		0	73	68					H,2,1					-21	35*	38	
			4	65	40		H,1,4						H,2,15		-25	37*	-31	
-1	122	123	-6	166	177				-4	29*	11				-27	10*	45	
3	107	-111	-12	72	-68	15	9*	-20	-12	8*	-3	-4	10*	-9				
-5	76	68	-16	65	62	-19	33*	-34	18	9*	5	-8	53*	-53		H,3,11		
-7	55	-62				21	39*	-46	-18	43*	-52	-8	51*	-43				
-11	155	166		H,6,8					20	33*	-16	-14	40*	39	1	9*	-1	
-13	171	-174					H,1,5					-16	9*	2	-1	31*	56	
-15	79	-75	-2	107	-105					H,2,2		-22	10*	-24	5	33*	15	
-17	73	61	-4	76	-70	1	25*	-14							7	10*	-27	
			-6	183	-186	17	42*	-33	2	24*	-49		H,2,16		-13	9*	-11	
	H,5,10		-8	91	-82	-25	13*	-9	8	40*	-27				-19	27*	20	
			-10	99	-94				16	36*	-24	-8	51*	57				
1	108	107	-14	93	92		H,1,6		22	52*	52	-16	43*	-15		H,3,12		
-1	86	83							-22	28*	-32	-16	28*	42				
-3	81	81		H,6,9		9	37*	28	-24	31*	14	-20	28*	41	3	47*	16	
-9	64	-61				17	26*	39							-3	22*	19	
-15	81	-74	-6	84	-73	-21	35*	44		H,2,3			H,3,0		-13	42*	38	
-19	133	-143	-8	65	71				-4	6*	13	1	6*	20	-15	9*	32	
			-14	59	43		H,1,7		14	36*	13	17	33*	24	-19	9*	49	
	H,5,11								16	45*	-48	15	11*	-5	-23	38*	-21	
				H,7,0		1	8*	-26	-26	49*	-1				-25	24*	-41	
-1	141	-135				-3	7*	3					H,3,1			H,3,13		
-5	127	134	7	53	53	7	11*	-3										
-15	89	93				-13	26*	-14		H,2,4								
-17	89	-104		H,7,1		-19	38*	41				17	27*	12	-1	10*	-23	
-19	87	-120				-21	45*	3	4	39*	-44	15	44*	-48				
			-5	60	-48	-27	23*	-13	-8	7*	27	-21	34*	-20		H,3,14		
	H,5,12		-7	78	-56				-14	30*	-30							
							H,1,8		16	40*	-29		H,3,2		-3	10*	31	
-7	75	72		H,7,2					-22	9*	1				-7	42*	-35	
-9	148	149				9	47*	48				9	25*	-32	-9	18*	-11	
-11	109	116	3	78	-75	-17	28*	-31		H,2,5		-13	13*	1	-11	39*	-48	
-13	228	223	5	65	-37	-29	52*	-52				15	16*	-30	-13	28*	-1	
-15	58	-51	-7	100	107				-2	7*	-1	17	9*	13	-15	39*	-35	
							H,1,9		-18	14*	-18	-17	9*	11	-17	34*	18	
	H,6,0			H,7,3					-22	19*	-12	15	40*	-49	-21	41*	27	
						3	42*	-53				21	54*	58				
0	228	-233	3	175	-165	-3	8*	7		H,2,6		-21	30*	-31		H,3,15		
2	154	-151	-5	60	44	5	17*	14				-25	24*	-27				
4	71	-76	-7	59	-47	9	47*	-33	-16	25*	-7				-13	54*	-54	
6	114	-116				-9	19*	-46	-24	38*	30		H,3,3		-17	17*	1	
8	169	166		H,0,2		-17	25*	6	-26	19*	-17							
10	147	148				-19	8*	-2				-7	8*	17		H,4,0		
12	93	-86	-26	43*	-35	-29	37*	22		H,2,7		17	38*	32				
14	78	81										15	46*	-51	14	34*	-25	
				H,0,4			H,1,10		4	33*	-25							
	H,6,1								-6	31*	38		H,3,4			H,4,1		
			-24	9*	-2	-7	36*	28	-18	28*	-35							
0	100	-107	-26	22*	3	11	43*	-40				-7	8*	-1	-6	8*	-2	
2	69	80	-28	10*	4	-13	43*	31		H,2,8		9	39*	-42	10	26*	6	
-2	110	-109				-23	22*	23				-11	8*	-21	18	29*	-31	
4	59	-51		H,0,6		-27	8*	23	0	34*	-33	13	24*	-3	20	40*	37	
-4	129	136							2	41*	38	15	28*	-10				
6	80	82	6	41*	31		H,1,11		4	9*	-18	19	17*	-5		H,4,2		
-6	63	-49	-18	8*	5				-10	40*	20	-23	9*	-9				
8	61	-55				1	18*	-5	-12	8*	24				0	13*	-20	
10	121	-123		H,0,8		-1	8*	-11	14	10*	-8		H,3,5		12	34*	-18	
-10	71	-57				3	28*	14	-16	35*	-50				-16	9*	-43	
			4	28*	-24	-11	40*	41	-26	8*	10	1	43*	-35	-22	49*	-37	
	H,6,2		-28	10*	33	-19	34*	19	-28	10*	-11	8	31*	12				
						-21	25*	32				-5	38*	-46		H,4,3		
0	97	106		H,0,10		-29	51*	49		H,2,9		-13	8*	-21				
4	66	84										17	10*	8	-10	25*	-8	
-4	91	-92	6	48*	-46		H,1,12		12	10*	-23	-15	9*	41	16	35*	14	
8	89	86	-16	46*	-60				-20	32*	-27	-23	35*	18	-22	18*	-15	
-8	158	-165	-24	49*	-59	1	9*	-12	-24	9*	8	-25	55*	44				
10	65	51				-1	18*	-26	-26	12*	-2					H,4,4		
-10	64	43		H,0,12		3	37*	-39	-28	48*	-63		H,3,6					
12	65	66				5	23*	20							10	9*	30	
-12	61	-46	-6	9*	6	-13	39*	-33		H,2,10		-5	23*	-27	-16	47*	48	
-14	147	-136	-18	32*	-53	-27	28*	-42				11	20*	-32	-20	9*	-24	
			-20	26*	26				-2	8*	-33	-11	23*	35	-22	38*	-10	
	H,6,3		-22	48*	41		H,1,13		10	21*	-15	-13	28*	39	-24	24*	-24	
			-28	29*	4				-10	34*	23	15	54*	-53				
-2	83	-96				1	38*	-28	-16	42*	-46	-17	47*	45		H,4,5		
-4	124	-136		H,0,14		-1	39*	-27	-20	45*	40	-21	9*	4				
6	87	-80				5	10*	-23	-28	37*	30	-23	44*	-35	4	14*	-26	
-6	77	76	0	10*	12	-5	9*	12				-25	30*	-23	8	42*	32	
-8	62	59	-6	17*	28	-13	5*	-25		H,2,11					14	10*	5	
-14	92	-96	-18	23*	33	-23	9*	6					H,3,7		-14	9*	5	
						-27	23*	-35	-2	31*	7				-22	36*	48	
	H,6,4			H,0,16					4	36*	43	1	8*	-5				
							H,1,14		6	41*	39	13	19*	20		H,4,6		
2	152	146	-18	10*	28				-14	8*	-9	15	33*	32				
-2	226	225				-3	36*	-29	-18	38*	10	-17	46*	-47	-2	35*	-17	
4	73	-75		H,1,0		-5	19*	11	-22	49*	48	-23	28*	-26	-4	34*	-30	
-4	157	162				-11	5*	2	-28	10*	0	-25	19*	30	12	14*	-22	
6	192	-197	21	39*	-21	-13	18*	-11				-27	22*	-2	-12	40*	-14	
-6	106	104				-15	43*	52		H,2,12					-24	32*	-44	
-8	88	101		H,1,1		-19	44*	47					H,3,8					
-12	110	-114							4	10*	4					H,4,7		
-16	59	44	5	13*	-13		H,1,15		-4	47*	-58	5	45*	-57				
			-11	15*	-36				-8	43*	6	-7	13*	20	4	29*	-15	
	H,6,5		-15	16*	-3	-5	19*	-34	-14	9*	6	11	10*	-47	8	44*	28	
			19	9*	-12	-7	28*	11	-26	52*	44	13	46*	56	-10	19*	15	
2	85	-86	-19	9*	15	-15	37*	-18				-15	26*	-16	12	31*	31	
-2	59	47	21	29*	23	-21	25*	10		H,2,13					-12	30*	36	
4	123	129	23	46*	53	-23	5*	-19					H,3,9					
-6	118	120				-25	10*	-3	-6	48*	-30					H,4,8		
-8	137	-141		H,1,2					-8	43*	-13	-3	28*	16				
-12	76	71					H,1,16		-10	19*	10	11	37*	10	0	45*	41	
-16	68	78	13	21*	18				-22	28*	-12	-11	8*	8	2	26*	44	
			17	30*	-26	-9	9*	10	-24	10*	3	-21	45*	-52	-2	19*	-13	
	H,6,6		-17	20*	24	-17	42*	-44	-26	50*	-46				-4	24*	20	
			19	45*	-30	-21	35*	40										

Table 8. Continued.

H,4,8

-14	51*	-56	-8
-20	39*	-37	-10
-24	15*	42	-14
			-16

H,4,9

2	9*	-5	
-4	9*	-20	5
-8	25*	24	13
-18	44*	57	

H,4,10

			-7
2	35*	52	11
-4	15*	10	-13
-6	9*	-7	15
-10	9*	28	-15
-12	20*	-36	17
-24	10*	-19	-17
			-19

H,4,11

0	27*	-30	
2	24*	44	9
4	13*	2	-13
-12	9*	6	15
-14	9*	-8	-15
-20	34*	15	-17

H,4,12

-14	9*	1	-1
-18	32*	-28	-17
-20	10*	33	-21
-22	10*	-6	

H,4,13

			7
-2	29*	-40	-9
-8	42*	47	-13
-12	44*	-57	-15
-16	10*	-14	-17
-20	32*	30	-19
			-21

H,4,14

43*	-46	-5	
10*	-1	11	
24*	-24	13	
10*	5	-17	
		-19	
		-21	

H,5,0

30*	-30	
11*	-1	
		-1

H,5,1

			-7
43*	-49	9	
9*	-5	-9	
39*	53	-13	
18*	30	-19	
34*	17	-21	
50*	-45		
34*	31		
10*	25		
		-3	
		-7	
		-9	

H,5,2

29*	-12	-13	
9*	-39	-17	
10*	6	-19	
9*	-19	-21	
28*	32		

H,5,3

			1
9*	4	7	
9*	-9	-13	
54*	-41	-17	
		-21	

H,5,4

11*	-5		
38*	51	1	
9*	15	-3	
14*	11	5	
13*	-27	-9	
10*	9	-19	
10*	14	-21	

H,5,5

14*	-9	-5	
10*	-7	-7	
27*	41	-11	
36*	-51	-13	
32*	-2	-17	
10*	-26		

H,5,6

			-3
9*	1	-7	
14*	-42	-9	
9*	-6	-11	
45*	65	-13	
19*	-21		
41*	50		
26*	-17		
19*	-36	-5	

H,5,7

9*	-20	-8	
44*	50	12	
47*	69	-12	
51*	57	14	
9*	17	-14	
43*	-36		
55*	-57		

H,5,8

			2
			-2
9*	-17	6	
43*	26	-6	
35*	-20	-16	
24*	10		
10*	42		

H,5,9

			0
			2
27*	-28	4	
9*	-4	8	
17*	-8	10	
40*	-18	-10	
10*	-2	-12	
45*	60	-16	

H,5,10

32*	32	0	
35*	0	8	
8*	9	10	
38*	-7	-10	
15*	-46	-14	

H,5,11

43*	-20	0	
42*	52	-4	
33*	-34	6	
19*	-6	8	
16*	8	-10	
		-14	

H,5,12

10*	-11	
		0

H,6,1

			2
			-4
9*	21	6	
55*	55	-6	
21*	-42	-8	
10*	-11		
10*	0		

H,6,2

			2
			-2
37*	29	-4	
47*	-22	-8	
10*	39	-10	
25*	-31	-14	
32*	9		

H,6,3

			0
9*	-10	-12	
47*	29	-16	
9*	-5		
20*	16		
49*	-43		
33*	9	-2	
15*	-11	-4	
36*	21	-10	
		-12	

H,6,4

43*	45	1	
43*	35	3	
10*	-21	5	
22*	-27		
27*	-57		

H,6,5

		1	
		-1	
41*	32	3	
43*	-54	-3	
28*	46	5	
37*	24	7	
9*	-5		
38*	-38		

H,6,6

		1	
		-1	
40*	14	-3	
14*	-4	-5	
32*	-16	-9	
10*	9		
9*	1		
45*	40		

H,6,7

		1	
		-1	
43*	-3	-3	
39*	-32		
23*	30		
26*	30		
36*	-51	1	
28*	-33	-1	
		-3	
		-5	
		-7	

H,6,8

			0
20*	11		
52*	-31		
10*	-13		

H,6,9

			-3
			-5
			-7
10*	-17		
48*	16		
10*	-10		
36*	37		

H,7,0

		1	
		3	
		5	
8*	-2		
33*	8		
17*	-35		

H,7,1

		1	
		-1	
10*	-44	3	
46*	31	-3	
10*	25	5	
10*	-22	7	
46*	-39		
28*	17		

H,7,2

		1	
		-1	
29*	-40	-3	
10*	34	-5	
31*	14	-9	
15*	12		
10*	5		

H,7,3

		1	
		-1	
10*	-7	-3	
10*	-33	-5	
35*	-39	-9	
10*	-19		

H,7,4

		1	
		-1	
41*	-20	-3	
53*	26	-5	
34*	-42	-7	
40*	46		
32*	9		

H,7,5

		3	
		-3	
49*	55	-5	
31*	-38	-7	
10*	-10		

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