$(BIGH_2)(NO_3)_2$			
N1—H1*A*···O6^{i}	0.86 (3)	2.20 (3)	151 (3)
N1—H1*B*···O2^{ii}	0.91 (3)	2.05 (3)	154 (3)
N2—H2*A*···O5^{i}	0.94 (3)	2.00 (2)	170 (2)
N2—H2*B*···O4^{iii}	0.87 (3)	2.09 (3)	167 (2)
N3—H3*A*···O3^{ii}	0.93 (3)	1.83 (3)	168 (3)
N4—H4*A*···O3^{iii}	0.94 (3)	2.04 (4)	157 (3)
N4—H4*B*···O4^{iv}	0.92 (4)	2.11 (4)	155 (2)
N5—H5*A*···O1^{i}	0.86 (3)	2.20 (2)	150 (3)
N5—H5*B*···O6^{v}	0.93 (3)	2.17 (3)	136 (2)

Symmetry codes: (i) $\frac{1}{2}+x, 2-y, z$; (ii) $\frac{1}{2}-x, y, z-\frac{1}{2}$; (iii) $\frac{1}{2}+x, 1-y, z$; (iv) $1-x, 1-y, z-\frac{1}{2}$; (v) $1-x, 1-y, \frac{1}{2}+z$.

Preliminary examinations and intensity data collection were carried out using an Enraf–Nonius CAD-4 diffractometer. Backgrounds were obtained from analysis of the scan profiles (Blessing, Coppens & Becker, 1974). Both structures were solved by direct methods and all atoms, including H atoms, refined cleanly.

For both compounds, data collection: CAD-4 software (Enraf–Nonius, 1977); cell refinement: CAD-4 software; data reduction: *MolEN PROCESS* (Fair, 1990); program(s) used to solve structures: *MULTAN*80 (Main *et al.*, 1980); program(s) used to refine structures: *MolEN LSFM*; molecular graphics: *CAChe* (CAChe Scientific, 1993); software used to prepare material for publication: *MolEN CIF IN*.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BK1041). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5,6,7,8-Tetrahydro-2,3-diphenyl-2aλ^4-thia-2,3,4a,8a-tetraazacyclopent[*cd*]azulene-1[2*H*],4[3*H*]-diselone

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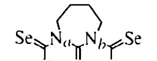
Abstract

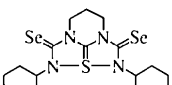
The title compound, $C_{19}H_{18}N_4SSe_2$, crystallizes in the $P\bar{1}$ space group with two molecules in the asymmetric unit. In both molecules, the chemically equivalent halves are identical within experimental error, except for conformational differences which arise from intermolecular interactions between the flexible seven-membered rings and surrounding Se atoms. The central triheterapentalene system is almost planar, with individual ring conformations consistent with three regions of conjugation separated by single bonds. The N—S bond lengths, stretched beyond the standard for a single bond, imply a three-centre interaction.

Comment

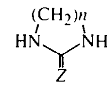
We have shown (Billing, Ferg, Lai, Levendis & Reid, 1993) that reaction of the cyclic thioureas [(3); *n* = 2, 3, 4, *Z* = S] and selenoureas [(3); *n* = 2, 3, 4, *Z* = Se] with 2,4-dinitrobenzyl chloride followed by deprotonation of the resulting salts with aqueous sodium carbonate yields the bases [(4); *n* = 2, 3, 4, *Z* = S, Se]. Treatment of the bases [(4); *n* = 2, 3, *Z* = S, Se] with heterocumulenes *R*NC*X* (*X* = S, Se) affords the triheterapentalenes containing the *X*—*Z*—*X* sequence (5) or triheterapentalenes (6) containing the N—*Z*—N sequence (Lai, 1992), depending on the bridge between the N_a and N_b atoms. To investigate further the influence of the bridge between the N_a and N_b atoms on the preferred heteroatom sequence (X—Z—X or N—Z—N), the base [(4); $n = 4$, Z = S] was treated with phenyl isoselenocyanate. The reaction product is shown to contain structure (1) on the basis of an X-ray single-crystal structure determination, with two molecules A and B in the asymmetric unit.



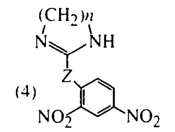
(1)



(2)

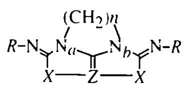


(3)

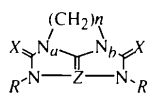


(4)

(3), (4) $n = 2, 3, 4$ Z = S, Se

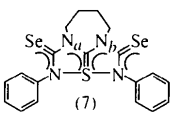


(5)



(6)

(5), (6) Z = S, Se X = S, Se



(7)

Due to the symmetrical connectivity of the molecule, the two halves of the molecule, separated by the bisector of the C(5)—C(6) bond through C(2) and S(1), are expected to be identical and any differences not of the conformational type are considered unlikely. This is confirmed by the individual and average values of bond lengths and angles over both halves, and over the two molecules, which were calculated for further discussion (see Fig. 1). The averaged bond lengths are shown in the right half of the diagram and bond angles on the left. Pairs of values shown around some bonds are the torsion angles for the two molecules, the upper values referring to molecule A. The following reference bond lengths, with e.s.d.'s of 0.01 Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987) were accepted as standards in order to assess the degree of delocalization within the heterocyclic system: C═S 1.63, C—S 1.65, C═N 1.28, C—N 1.48, C═Se 1.74, C—Se 1.97 and N—S 1.74 Å.

In terms of these criteria, one recognizes three islands of conjugation, (7), separated by single C—N bonds, *i.e.* Se(1)—C(1)—N(1), N(2)—C(2)[═S(1)]—N(3) and Se(2)—C(3)—N(4). The long N—S bonds indicate a three-centre interaction of the sort we have previously described for compound (2) (Billing, Boeyens, Denner, Hellyar, Lai, Matthee & Reid, 1992).

To substantiate this interpretation, the least-squares mean plane through the triheterapentalene core was calculated. The following deviations (Å) from the plane are observed: for molecule A N(1) 0.033 (2), C(1) −0.024 (2), N(2) −0.022 (2), C(2) 0.000 (3), N(3) 0.037 (2), C(3) −0.001 (2), N(4) −0.032 (2), S(1) 0.009 (1); for molecule B N(1) 0.031 (2), C(1) 0.029 (2), N(2) −0.055 (2), C(2) −0.022 (3), N(3) 0.037 (2), C(3) 0.025 (2), N(4) −0.022 (2), S(1) −0.022 (1). The system is essentially flat and the puckers of individual rings are likewise small, but consistent, with twist conformations shown to predominate in terms of quantitative analysis of ring pucker (Evans & Boeyens, 1989).

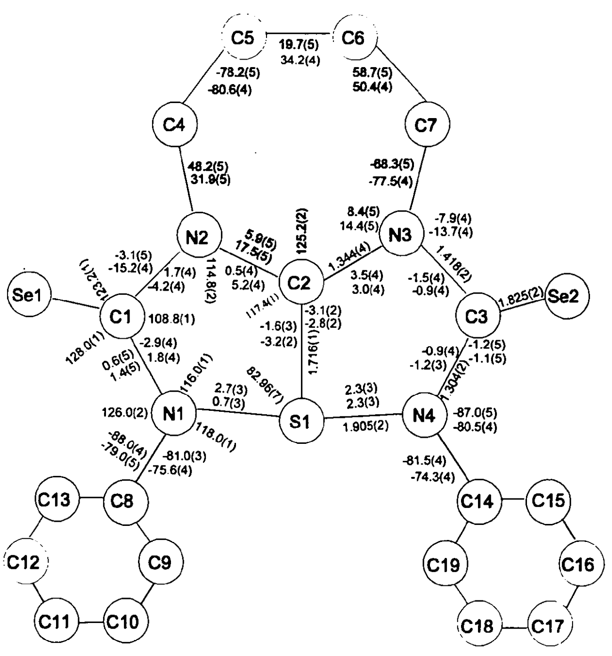


Fig. 1. Schematic drawing of (1). Molecular parameters (Å, °) are averaged over chemically equivalent bonds in molecules A and B. Upper values refer to molecule A.

The major difference between the two independent molecules is in the conformation of the seven-membered rings and the intramolecular orientation of these rings with respect to the Se atoms. The differences, also reflected by the endocyclic torsion angles shown in Fig. 1, are most probably due to intermolecular interactions. Some of the shortest non-bonded contacts occur between the H atoms attached to the ring atoms C(4)–C(7) and the Se atoms of neighbouring molecules. These contacts are not symmetrical between the two independent molecules, which suggests that the conformational differences represent slight distortions which promote closest packing.

The conformation of the A ring [N(2)—C(4)—C(5)—C(6)—C(7)—N(3)—C(2)] resembles that of the boat-sofa $BS\bar{7}$ (Boessenkool & Boeyens, 1980) but is actually a blend of four primitive forms, formally represented (Evans & Boeyens, 1989) by

$$\chi(A) = 0.11\,(24) + 0.62\,(23) + 0.06\,(12) + 0.21\,(13),$$

where the angular values in brackets correspond to

$k\pi/14$. The primitive forms involved are

$$0.11\ (B5) + 0.62\ (TB6) + 0.06\ (C\bar{7}) + 0.21\ (TC4).$$

In the same terminology, for the B ring

$$\chi(B) = 0.63\,(24) + 0.13\,(25) + 0.24\,(13),$$

$$i.e.\ 0.63\ (B5) + 0.13\ (TB\bar{4}) + 0.24\ (TC4).$$

Views of molecules A and B are shown in Fig. 2 and a stereoview of molecule B is shown in Fig. 3.

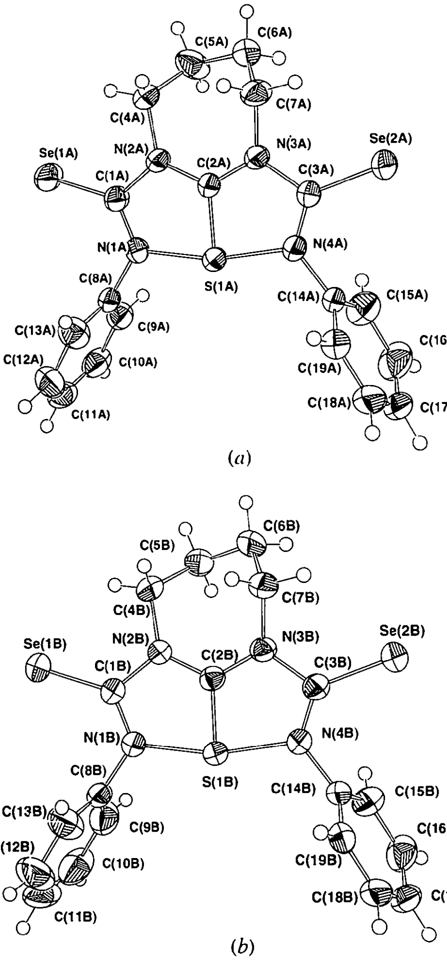


Fig. 2. View of (1) showing the atom-labelling scheme for (a) molecule A and (b) molecule B. Displacement ellipsoids are plotted at the 50% probability level.

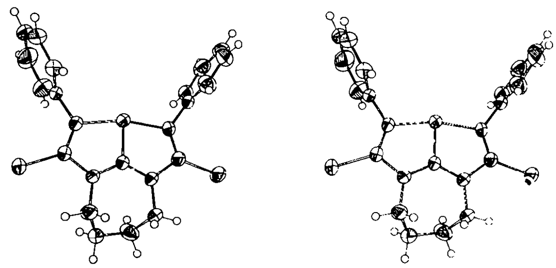


Fig. 3. Stereoscopic drawing of the conformation of molecule B.

Experimental

The title compound was recrystallized from MeCN–CH_2Cl_2 (2:1) as clear crystals, m.p. 419–420 K.

Crystal data

$C_{19}H_{18}N_4SSe_2$
$M_r = 492.35$
Triclinic
$P\bar{1}$
$a = 9.395\,(7)$ Å
$b = 14.373\,(8)$ Å
$c = 14.530\,(5)$ Å
$\alpha = 89.11\,(4)°$
$\beta = 88.27\,(4)°$
$\gamma = 86.79\,(4)°$
$V = 1958\,(2)$ Å^3
$Z = 4$
$D_x = 1.670$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–20°$
$\mu = 3.894$ mm^{-1}
$T = 293\,(2)$ K
Tabular
1.1 × 0.7 × 0.5 mm
Light yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: *ABSORB* (*Xtal*3.2; Hall, Flack & Stewart, 1992)
$T_{\min} = 0.985$, $T_{\max} = 0.999$
9942 measured reflections
8411 independent reflections

5641 observed reflections [$F > 4\sigma(F)$]
$R_{\text{int}} = 0.02$
$\theta_{\max} = 30°$
$h = -1 \rightarrow 11$
$k = -18 \rightarrow 18$
$l = -20 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity variation: random

Refinement

Refinement on F^2
$R(F) = 0.0791$
$wR(F^2) = 0.1052$
$S = 1.070$
8411 reflections
471 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0610P)^2]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$

$(\Delta/\sigma)_{\max} = 0.413$
$\Delta\rho_{\max} = 1.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.80$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Se(1A)	0.1869 (1)	0.8489 (1)	1.0261 (1)	0.066 (1)
Se(2A)	0.2019 (1)	0.7106 (1)	0.4882 (1)	0.053 (1)
S(1A)	0.4121 (1)	0.7627 (1)	0.7586 (1)	0.038 (1)
N(1A)	0.3838 (3)	0.7999 (2)	0.8830 (2)	0.042 (1)
N(2A)	0.1611 (3)	0.8053 (2)	0.8357 (2)	0.037 (1)
N(3A)	0.1674 (3)	0.7670 (2)	0.6760 (2)	0.036 (1)
N(4A)	0.3914 (3)	0.7289 (2)	0.6333 (2)	0.040 (1)
C(1A)	0.2516 (3)	0.8161 (2)	0.9108 (2)	0.039 (1)
C(2A)	0.2297 (3)	0.7794 (2)	0.7566 (2)	0.034 (1)
C(3A)	0.2622 (3)	0.7359 (2)	0.6030 (2)	0.036 (1)
C(4A)	0.0039 (4)	0.8162 (3)	0.8494 (3)	0.057 (1)
C(5A)	−0.0698 (4)	0.7343 (3)	0.8149 (3)	0.061 (1)
C(6A)	−0.0844 (4)	0.7332 (3)	0.7131 (3)	0.058 (1)
C(7A)	0.0157 (4)	0.7924 (3)	0.6604 (2)	0.054 (1)
C(8A)	0.5068 (3)	0.8038 (2)	0.9377 (2)	0.039 (1)
C(9A)	0.5648 (4)	0.7226 (3)	0.9762 (2)	0.047 (1)
C(10A)	0.6879 (4)	0.7253 (3)	1.0253 (3)	0.059 (1)

C(11*A*)	0.7510 (4)	0.8070 (4)	1.0359 (3)	0.063 (1)
C(12*A*)	0.6960 (5)	0.8862 (3)	0.9975 (3)	0.069 (1)
C(13*A*)	0.5718 (4)	0.8860 (3)	0.9470 (3)	0.057 (1)
C(14*A*)	0.5180 (3)	0.6976 (2)	0.5832 (2)	0.039 (1)
C(15*A*)	0.5566 (5)	0.6054 (3)	0.5810 (3)	0.063 (1)
C(16*A*)	0.6883 (5)	0.5758 (4)	0.5410 (3)	0.078 (1)
C(17*A*)	0.7760 (4)	0.6388 (4)	0.5048 (3)	0.074 (1)
C(18*A*)	0.7367 (4)	0.7305 (4)	0.5058 (3)	0.068 (1)
C(19*A*)	0.6064 (4)	0.7612 (3)	0.5457 (3)	0.054 (1)
Se(1*B*)	0.3113 (1)	0.4731 (1)	0.1585 (1)	0.063 (1)
Se(2*B*)	0.2800 (1)	1.0140 (1)	0.3003 (1)	0.053 (1)
S(1*B*)	0.0761 (1)	0.7409 (1)	0.2516 (1)	0.035 (1)
N(1*B*)	0.1081 (3)	0.6151 (2)	0.2143 (2)	0.041 (1)
N(2*B*)	0.3290 (3)	0.6604 (2)	0.2179 (2)	0.036 (1)
N(3*B*)	0.3183 (3)	0.8221 (2)	0.2525 (2)	0.034 (1)
N(4*B*)	0.0938 (3)	0.8682 (2)	0.2823 (2)	0.037 (1)
C(1*B*)	0.2415 (3)	0.5869 (2)	0.1990 (2)	0.039 (1)
C(2*B*)	0.2580 (3)	0.7411 (2)	0.2402 (2)	0.033 (1)
C(3*B*)	0.2223 (3)	0.8975 (2)	0.2782 (2)	0.036 (1)
C(4*B*)	0.4847 (3)	0.6397 (2)	0.2276 (3)	0.048 (1)
C(5*B*)	0.5443 (4)	0.6893 (3)	0.3082 (3)	0.050 (1)
C(6*B*)	0.5730 (4)	0.7902 (2)	0.2866 (3)	0.050 (1)
C(7*B*)	0.4665 (3)	0.8360 (2)	0.2219 (2)	0.043 (1)
C(8*B*)	−0.0112 (3)	0.5595 (2)	0.2035 (3)	0.044 (1)
C(9*B*)	−0.0797 (4)	0.5259 (3)	0.2805 (3)	0.067 (1)
C(10*B*)	−0.1995 (5)	0.4770 (3)	0.2714 (5)	0.095 (2)
C(11*B*)	−0.2516 (5)	0.4608 (3)	0.1883 (6)	0.101 (2)
C(12*B*)	−0.1820 (6)	0.4946 (3)	0.1110 (5)	0.093 (2)
C(13*B*)	−0.0629 (4)	0.5446 (3)	0.1192 (3)	0.066 (1)
C(14*B*)	−0.0326 (3)	0.9229 (2)	0.3072 (2)	0.037 (1)
C(15*B*)	−0.0677 (4)	0.9386 (3)	0.3993 (3)	0.050 (1)
C(16*B*)	−0.1971 (5)	0.9857 (3)	0.4215 (3)	0.065 (1)
C(17*B*)	−0.2890 (4)	1.0136 (3)	0.3539 (4)	0.062 (1)
C(18*B*)	−0.2536 (4)	0.9965 (3)	0.2633 (3)	0.063 (1)
C(19*B*)	−0.1238 (4)	0.9515 (2)	0.2391 (3)	0.049 (1)

Table 2. *Selected geometric parameters (Å, °)*

Se(1*A*)—C(1*A*)	1.824 (3)	Se(1*B*)—C(1*B*)	1.826 (3)
Se(2*A*)—C(3*A*)	1.826 (3)	Se(2*B*)—C(3*B*)	1.825 (3)
S(1*A*)—C(2*A*)	1.719 (3)	S(1*B*)—C(2*B*)	1.712 (3)
S(1*A*)—N(1*A*)	1.899 (3)	S(1*B*)—N(1*B*)	1.901 (3)
S(1*A*)—N(4*A*)	1.910 (3)	S(1*B*)—N(4*B*)	1.907 (3)
N(1*A*)—C(1*A*)	1.304 (4)	N(1*B*)—C(1*B*)	1.309 (4)
N(2*A*)—C(2*A*)	1.348 (4)	N(2*B*)—C(2*B*)	1.344 (4)
N(2*A*)—C(1*A*)	1.419 (4)	N(2*B*)—C(1*B*)	1.411 (4)
N(3*A*)—C(2*A*)	1.344 (4)	N(3*B*)—C(2*B*)	1.340 (4)
N(3*A*)—C(3*A*)	1.425 (4)	N(3*B*)—C(3*B*)	1.418 (4)
N(4*A*)—C(3*A*)	1.302 (4)	N(4*B*)—C(3*B*)	1.300 (4)
C(2*A*)—S(1*A*)—N(1*A*)	83.1 (1)	C(2*B*)—S(1*B*)—N(1*B*)	82.9 (1)
C(2*A*)—S(1*A*)—N(4*A*)	83.0 (1)	C(2*B*)—S(1*B*)—N(4*B*)	82.8 (1)
N(1*A*)—S(1*A*)—N(4*A*)	166.1 (1)	N(1*B*)—S(1*B*)—N(4*B*)	165.6 (1)
C(1*A*)—N(1*A*)—S(1*A*)	116.0 (2)	C(1*B*)—N(1*B*)—S(1*B*)	115.9 (2)
C(2*A*)—N(2*A*)—C(1*A*)	114.5 (3)	C(2*B*)—N(2*B*)—C(1*B*)	114.7 (3)
C(2*A*)—N(3*A*)—C(3*A*)	114.8 (3)	C(2*B*)—N(3*B*)—C(3*B*)	115.0 (3)
C(3*A*)—N(4*A*)—S(1*A*)	116.0 (2)	C(3*B*)—N(4*B*)—S(1*B*)	116.1 (2)
C(14*A*)—N(4*A*)—S(1*A*)	117.1 (2)	N(1*B*)—C(1*B*)—N(2*B*)	108.8 (3)
N(1*A*)—C(1*A*)—N(2*A*)	109.0 (3)	N(1*B*)—C(1*B*)—Se(1*B*)	127.9 (2)
N(1*A*)—C(1*A*)—Se(1*A*)	127.3 (2)	N(2*B*)—C(1*B*)—Se(1*B*)	123.3 (2)
N(2*A*)—C(1*A*)—Se(1*A*)	123.7 (2)	N(3*B*)—C(2*B*)—N(2*B*)	125.0 (3)
N(3*A*)—C(2*A*)—N(2*A*)	125.4 (3)	N(3*B*)—C(2*B*)—S(1*B*)	117.4 (2)
N(3*A*)—C(2*A*)—S(1*A*)	117.2 (2)	N(2*B*)—C(2*B*)—S(1*B*)	117.5 (2)
N(2*A*)—C(2*A*)—S(1*A*)	117.3 (2)	N(4*B*)—C(3*B*)—N(3*B*)	108.6 (3)
N(4*A*)—C(3*A*)—N(3*A*)	108.7 (3)	N(4*B*)—C(3*B*)—Se(2*B*)	128.5 (2)
N(4*A*)—C(3*A*)—Se(2*A*)	128.3 (2)	N(3*B*)—C(3*B*)—Se(2*B*)	122.9 (2)
N(3*A*)—C(3*A*)—Sc(2*A*)	122.9 (2)		

Data collection and data reduction were carried out using standard Enraf–Nonius routines. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985) and refined using *SHELXL*93 (Sheldrick, 1993). H atoms were placed in geometrically calculated positions and allowed to refine, riding on the atoms to which they are attached, with a common isotropic displacement parameter. The labelled structure diagram was produced using *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1038). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Three Protected Tetrapeptides

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Abstract

The structures of three protected tetrapeptides, containing the Boc-Gly-Gly-Phe-*X*-OMe chain, *tert*-butoxycarbonyl-glycy-glycl-phenylalanine-leucine methyl ester dihydrate, Boc-Gly-Gly-L-Phe-D-Leu-OMe, $C_{25}H_{38}N_4O_7.2H_2O$, *tert*-butoxycarbonyl-glycy-glycl-phenylalanine-methionine methyl ester dihydrate, Boc-Gly-Gly-L-Phe-D-Met-OMe, $C_{24}H_{36}N_4O_7S.2H_2O$ and *tert*-butoxycarbonyl-glycy-glycl-phenylalanine-norleucine methyl ester dihydrate, Boc-Gly-Gly-D-Phe-L-Nle-OMe, $C_{25}H_{38}N_4O_7.2H_2O$, are described. The three molecules have the same conformation of the tetrapeptide chain and display the same packing, consisting of couples