Taylor, 1987). The S(3)—C(2) bond of 1·714 (4) Å is longer than the C=S bond of 1·683 (1) Å in the substituted thione 4-phenylthiazole-2(3H)-thione (Nalini & Desiraju, 1989), and is comparable to the S(1)—C(1) and S(2)—C(1) bonds of 1·715 (4) and 1·717 (3) Å respectively, but is significantly shorter than the S(1)—C(2) and S(2)—C(3) bonds of 1·777 (3) and 1·745 (3) Å respectively. The short S(3)—C(2) bond can be viewed as a single bond with the S atom carrying a negative charge so that C(2) is positive relative to S(3). The comparable and short S(1)—C(1) and S(2)—C(1) bonds can be viewed as a positive charge in the pentatomic ring close to C(1), so that C(1) is positive relative to the S(1) and S(2) atoms. The pentatomic ring system is nearly planar and the torsion angles involving the ring system are close to 0 and 180°. All the features of the pentatomic heterocyclic system in this compound agree very well with those in 2-morpholino-5-propyl-1,3-dithio-lium-4-thiolate (Cheung, Galembeck, Miller, de Oliveira, Pereira & Simas, 1992). The C(1)—N bridging distance between the two ring systems is 1·323 (4) Å. The piperidyl ring has a chair conformation with N and C(7) deviating −0·500 (3) and

0·708 (4) Å respectively from the plane through C(5), C(6), C(8) and C(9). The two C_{sp^3}—N_{sp^3} bonds are 1·481 (5) and 1·480 (5) Å. The two C_{sp^3}—C_{sp^3} bonds [1·525 (7) and 1·523 (7) Å] farther away from the N atom are longer than the other two [1·516 (5) and 1·517 (5) Å] although the differences are not significant. All six bond angles are close to 109·5°.

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Stereochemical Studies. 124.* Structures of (I) *cis*- and (II) *trans*-Decahydroquinazoline-2-spiro-1'-cyclohexan-4-one

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Abstract. $C_{13}H_{22}N_2O$, $M_r = 222·32$. The title compound has two conformations, *cis* (I) and *trans* (II). Compound (I): monoclinic, $P2_1/c$, $a = 10·859$ (2), $b = 10·745$ (3), $c = 10·975$ (2) Å, $\beta = 101·13$ (3)°, $V = 1256·5$ (3) Å^3, $Z = 4$, $D_x = 1·175$ Mg m^{-3}, m.p. = 428–429 K, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·070$ mm^{-1}, $F(000) = 488$, $T = 293$ K, final $R = 0·058$ for 1735 reflections with $I > 2·5\sigma(I)$. Compound (II): triclinic, $P\bar{1}$, $a = 11·836$ (1), $b = 9·879$ (1), $c = 5·726$ (2) Å, $\alpha = 101·23$ (1), $\beta = 103·08$ (1), $\gamma = 94·02$ (1)°, $V = 635·1$ (1) Å^3, $Z = 2$, $D_x = 1·162$ Mg m^{-3}, m.p. = 471–472 K, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0·505$ mm^{-1}, $F(000) = 244$, $T = 293$ K, final $R = 0·044$ for 1792 reflections with $I > 1·5\sigma(I)$. The molecules in the two compounds have the expected bond lengths and angles and form dimers connected through N—H···O hydrogen bonds with the following lengths and angles: N3···O11 = 2·868 (3), H3···O11 = 2·056 (32) Å, ∠N3—H3···O11 = 165·0 (31)° in compound (I), and 2·861 (2), 1·979 (24) Å, 173·3 (21)°, respectively, in compound (II). The conformation of the pyrimidine ring is $^1H_6$ in both compounds.

Introduction. The quinazolin-4-one moiety is present in a number of different alkaloids and the chemistry and pharmacology of quinazolinones have been thoroughly studied (Johne, 1984), but the fully saturated analogues (Armarego, 1979) have received little attention. We have recently prepared a number of differently substituted fully saturated quinazolin-4-ones (Fülöp, Bernáth, Pihlaja, Mattinen, Argay & Kálmán, 1987; Fülöp, Pihlaja, Mattinen & Bernáth, 1987; Pihlaja, Fülöp, Mattinen & Bernáth, 1987). As a continuation of a project involving the synthesis and stereochemical investigation of saturated heterocycles, we now report the results of X-ray analyses of the title compounds.

Experimental. Compounds (I) and (II) were synthesized as described earlier (Pihlaja et al., 1987) from cis- and trans-2-aminocyclohexanecarboxamide and cyclohexanone. Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation for compound (I), Philips PW1100 diffractometer with graphite-monochromated Cu $K\alpha$ radiation for compound (II). Unit-cell parameters from 25 reflections in the range $10 < \theta < 15°$ (I) and 16 reflections in the range $15 < \theta < 18°$ (II). Colourless prismatic crystal, $0.38 \times 0.41 \times 0.40$ mm (I) and colourless plate with approximate dimensions $0.10 \times 0.30 \times 0.81$ mm (II). Independent reflections were measured with the $\omega-2\theta$ scanning technique, scan width 1.1 (I) and $1.5°$ (II), scan speed 0.06 (I) and $0.05° \, s^{-1}$ (II). 3200 reflections were collected for (I) with h: -14 to 14, k: -14 to 14, l: 0 to 14, and $2\theta_{max} = 56°$. 2026 reflections were measured for (II) with h: -14 to 13, k: -11 to 11, l: 0 to 6, and $2\theta_{max} = 140°$. Subsequent calculations involved 1735 (I) and 1792 (II) intensity data with $I > 2.5\sigma(I)$ for (I) and $I > 1.5\sigma(I)$ for (II). Three standard reflections, no intensity variation. Neither absorption nor extinction corrections were applied. Structures were solved by SHELX76 (Sheldrick, 1976) and SHELXS86 (Sheldrick, 1986). Full-matrix refinement on $\Sigma w(\Delta F)^2$, anisotropic non-H atoms. Positions of H atoms were generated from assumed geometries, checked in Fourier maps and refined, for compound (I) with a final overall $U_{iso} = 0.067$ (2) Å^2, while for (II) they were refined isotropically. Final $R = 0.058$, $wR = 0.066$, $w = 1.1000 \times [\sigma^2(F) + 0.002035(F)^2]^{-1}$ for 212 parameters (I), and $R = 0.044$, $wR = 0.048$, $w = 1/\sigma^2(F)$ for 233 parameters (II). Difference Fourier maps showed the following peaks: (I) maximum 0.54, minimum -0.24 e Å^{-3}; (II) maximum 0.20, minimum -0.25 e Å^{-3}. The higher value of the peak in compound (I) can be attributed to the lone electron pair of the N1 atom. Maximum Δ/σ was <0.321 (I) and <0.035 (II). Scattering factors as in SHELX76. All calculations for (I) were performed on an IBM 43/41 computer and for (II) on a PC/AT computer.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\times 10^4$) *with e.s.d.'s in parentheses*

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

|	x	y	z	U_{eq} (Å^2)
(I)				
N1	7848 (2)	241 (2)	8254 (2)	396 (5)
C2	7447 (2)	822 (2)	7045 (2)	353 (5)
N3	6235 (2)	296 (2)	6397 (2)	410 (5)
C4	5478 (2)	-466 (2)	6846 (2)	411 (5)
C5	5873 (2)	-905 (3)	8159 (2)	460 (6)
C6	6834 (3)	-11 (3)	8920 (2)	497 (6)
C7	6342 (3)	-2210 (3)	8159 (3)	546 (7)
C8	6805 (4)	-2697 (3)	9485 (3)	682 (8)
C9	7818 (3)	-1851 (3)	10167 (3)	668 (9)
C10	7351 (4)	-543 (3)	10224 (3)	637 (8)
O11	4487 (2)	-859 (2)	6197 (2)	579 (5)
C12	7288 (3)	2228 (2)	7192 (3)	464 (6)
C13	8537 (3)	2871 (3)	7672 (3)	556 (7)
C14	9474 (3)	2616 (3)	6858 (3)	569 (7)
C15	9661 (3)	1243 (3)	6725 (3)	530 (7)
C16	8423 (2)	581 (3)	6235 (2)	422 (6)
(II)				
N1	2607 (1)	5145 (1)	847 (3)	409 (3)
C2	2319 (1)	5891 (2)	3031 (3)	361 (3)
N3	1219 (1)	5225 (1)	3376 (3)	434 (3)
C4	694 (1)	3936 (2)	2293 (3)	406 (4)
C5	1169 (1)	3112 (2)	309 (3)	399 (4)
C6	2439 (1)	3637 (2)	555 (3)	380 (4)
C7	1002 (2)	1552 (2)	143 (5)	572 (6)
C8	1440 (2)	782 (2)	-1984 (5)	661 (6)
C9	2696 (2)	1316 (2)	-1834 (5)	638 (6)
C10	2856 (2)	2888 (2)	-1608 (4)	528 (5)
O11	-181 (1)	3454 (1)	2859 (3)	587 (3)
C12	3294 (1)	5933 (2)	5320 (3)	438 (4)
C13	4429 (2)	6737 (2)	5284 (5)	566 (5)
C14	4253 (2)	8215 (2)	5017 (5)	614 (5)
C15	3285 (2)	8214 (2)	2774 (4)	570 (5)
C16	2149 (2)	7389 (2)	2799 (4)	483 (4)

(I)

(II)
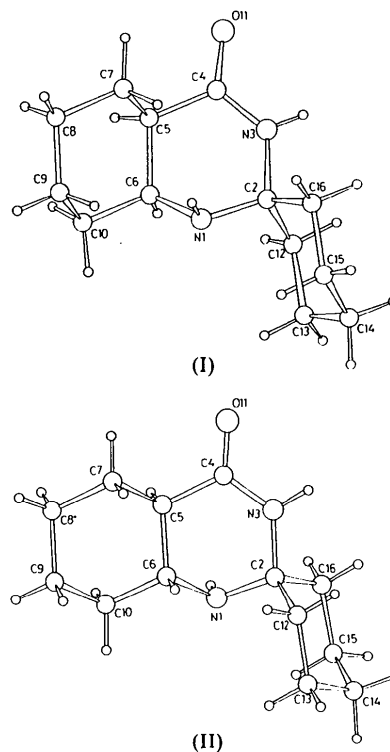
Fig. 1. Perspective views of the molecules showing atomic numbering. The H atoms are shown but not labelled.

Table 2. *Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses*

|	(I)	(II)		(I)	(II)
N1—C2	1·455 (3)	1·447 (2)	C5—C7	1·492 (5)	1·521 (3)
N1—C6	1·460 (4)	1·461 (2)	C6—C10	1·543 (4)	1·515 (3)
C2—N3	1·482 (3)	1·488 (2)	C7—C8	1·537 (4)	1·520 (4)
C2—C12	1·533 (3)	1·529 (2)	C8—C9	1·510 (5)	1·520 (3)
C2—C16	1·531 (3)	1·534 (3)	C9—C10	1·500 (5)	1·528 (3)
N3—C4	1·321 (3)	1·341 (2)	C12—C13	1·523 (4)	1·518 (3)
C4—C5	1·498 (3)	1·504 (2)	C13—C14	1·503 (5)	1·521 (3)
C4—O11	1·244 (2)	1·244 (2)	C14—C15	1·500 (5)	1·515 (3)
C5—C6	1·540 (4)	1·521 (2)	C15—C16	1·524 (4)	1·527 (3)
C2—N1—C6	114·5 (2)	113·3 (1)	C6—C5—C7	113·0 (2)	111·5 (2)
N1—C2—N3	110·8 (2)	111·0 (1)	N1—C6—C5	110·0 (2)	111·4 (1)
N1—C2—C12	110·3 (2)	111·0 (1)	N1—C6—C10	111·0 (2)	112·1 (1)
N1—C2—C16	109·8 (2)	109·6 (1)	C5—C6—C10	111·0 (2)	110·2 (1)
N3—C2—C12	108·6 (2)	108·3 (1)	C5—C7—C8	111·7 (3)	110·5 (2)
N3—C2—C16	107·9 (2)	108·3 (1)	C7—C8—C9	109·8 (3)	111·9 (2)
C12—C2—C16	109·4 (2)	108·5 (1)	C8—C9—C10	111·4 (3)	111·7 (2)
C2—N3—C4	128·1 (2)	126·8 (1)	C6—C10—C9	112·0 (3)	111·0 (2)
N3—C4—C5	118·1 (2)	118·0 (1)	C2—C12—C13	111·9 (2)	112·6 (2)
N3—C4—O11	121·9 (2)	121·0 (2)	C12—C13—C14	111·8 (3)	111·1 (2)
C5—C4—O11	120·0 (2)	121·0 (2)	C13—C14—C15	110·9 (3)	110·7 (2)
C4—C5—C6	111·3 (2)	111·4 (1)	C14—C15—C16	111·5 (3)	112·0 (2)
C4—C5—C7	109·3 (2)	113·3 (2)	C2—C16—C15	112·2 (2)	111·9 (2)
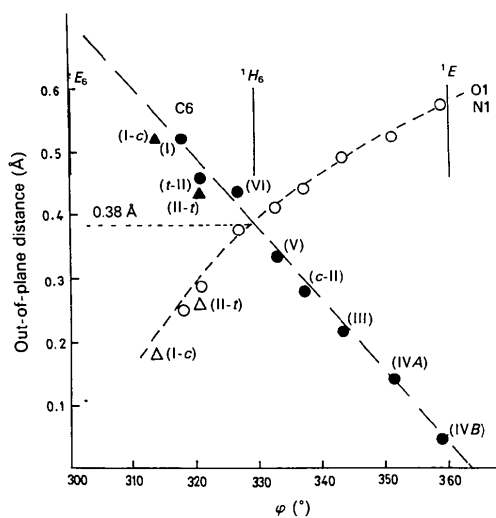
Fig. 2. Plots of the out-of-plane distances for C6 (full circles) and O1 (open circles) or N1 (full and open triangles for present study) against the puckering parameter φ in the range of the E_6 and ^1E canonical forms. The out-of-plane distances refer to the least-squares planes of ring atoms C2, N3, C4 and C5. (I-*c*), (II-*t*) (present study); (I), (*c*-II), (*t*-II) (Ribár *et al.*, 1989); (III) (Argay, Kálmán, Ribár, Lazar & Bernáth, 1980*a*); (IV) (Argay, Kálmán, Ribár, Fülöp & Bernáth, 1982); (V) (Kapor, Ribár, Argay, Kálmán, Fülöp & Bernáth, 1985); (VI) (Argay, Kálmán, Ribár, Lazar & Bernáth, 1980*b*).

Discussion. The final atomic coordinates for non-H atoms are listed in Table 1.* Fig. 1 shows a perspective view of the molecules. The relevant bond lengths and bond angles are given in Table 2. Bond lengths

* 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 54368 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

involving H atoms lie within the range 0·798 (33)–1·119 (36) (I) and 0·886 (24)–1·055 (27) Å (II). The corresponding values for the majority of bond angles and distances are similar. Differences in the bond lengths in the six-membered ring C5...C10 of compound (I) with respect to those in compound (II), are probably the consequence of the influence of the lone electron pair of the N1 atom which, as a result of the *cis* junction, comes much closer to the electron cloud of the ring. The values of the torsion angles C7—C5—C6—N1 = − 73·2 (3) (I), − 176·9 (1)° (II), and C10—C6—C5—C4 = 173·5 (2) (I), − 174·3 (1)° (II) indicate that the *cis* junction in compound (I) involves axial N1 and equatorial C4 positions and the *trans* junction in (II) involves both equatorial N1 and C4 positions.
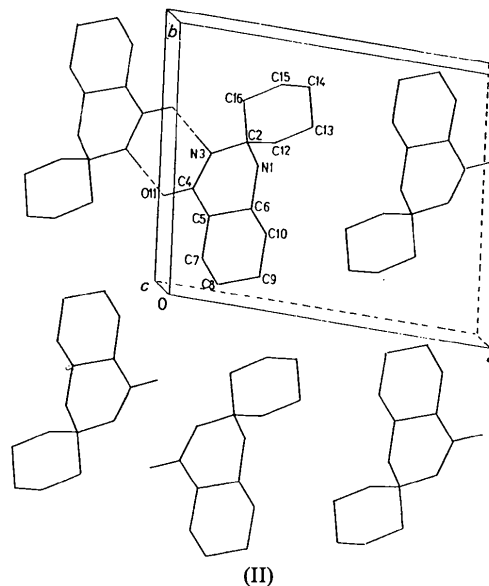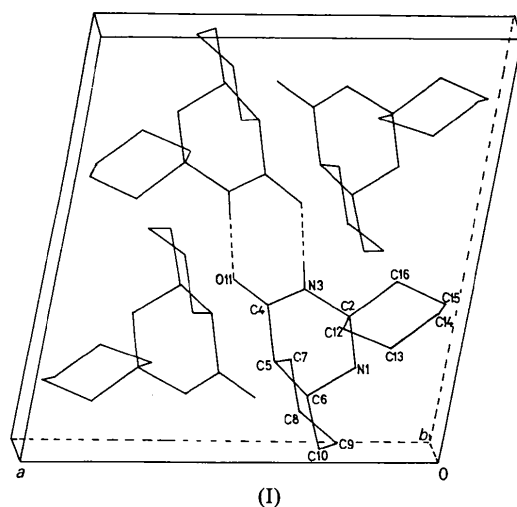

(I)

(II)

Fig. 3. Crystal packing diagrams for title compounds (I) and (II). Hydrogen bonds are indicated by dashed lines.

In both compounds the puckering parameters (Cremer & Pople, 1975) for the pyrimidine ring [Q = 0·464 (3) (I), 0·455 (2) Å (II); θ = 49·1 (4) (I), 44·0 (2)° (II); φ = 313·5 (5) (I), 320·2 (3)° (II)], the six-membered C5...C10 ring [Q = 0·557 (4) (I), 0·569 (2) Å (II); θ = 5·4 (4) (I), 2·7 (2)° (II)] and the spirocyclohexane C2—C12...C16 ring [Q = 0·562 (3) (I), 0·569 (2) Å (II); θ = 0·4 (3) (I), 1·5 (2)° (II)] indicate a half-chair conformation for the hetero ring and chair conformations for the two carbocyclic rings, with $^1H_6$, $^1C_4$ and $^1C_4$ conformational forms, respectively (Boeyens, 1978). The out-of-plane amplitudes (Å) for atoms N1 and C6, with respect to the least-squares planes of ring atoms C2, N3, C4, C5, are 0·178 (2) and $-0\cdot513$ (3) for (I) and 0·254 (1) and $-0\cdot430$ (1) for (II). In our previous study (Ribár, Kapor, Kálmán, Argay, Fülöp & Bernáth, 1989), we plotted the out-of-plane distances for C6 atom against the puckering parameter φ for a series of related compounds. Here, we reproduce this diagram (Fig. 2), with the points for the C6 and N1 atoms from this study added. (It is important to note that here the N1 atom has taken the role of O1 atom in the previously studied compounds.) One can see that the new values are in good agreement with plotted curves, indicating a similar conclusion, *i.e.* the pyrimidin-4-one moiety, like the 1,3-oxazin-4-one moiety (Ribár *et al.*, 1989), adopts a $^1H_6$ (Boeyens, 1978) half-chair conformation in both compounds.

The molecules of the compounds are connected through hydrogen bonds of N—H···O type to form dimers. The corresponding bond lengths and angles are H3···O11 = 2·056 (32) (I), 1·979 (24) Å (II),

N3···O11 = 2·868 (3) (I), 2·861 (2) Å (II), $\angle$N3—H3···O11 = 165·0 (31) (I), 173·3 (21)° (II). The molecular packing in the crystal is shown in Fig. 3.

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Structure of 3',5'-Di-O-acetyl-N⁴-methoxycytosine

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Abstract. $C_{14}H_{19}N_3O_7$, M_r = 341·32, orthorhombic, $P2_12_12_1$, a = 11·219 (8), b = 12·136 (7), c = 12·324 (7) Å, V = 1678 (3) Å³, Z = 4, D_x = 1·35, D_m = 1·34 (2) g cm⁻³, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·18 cm⁻¹, $F(000)$ = 720, T = 292 K, R = 0·075 for 1096 unique observed diffractometer data [$I \geq 2\cdot0\sigma(I)$]. The modified base N^4-methoxycytosine is present in the imino tautomeric form, with the methoxy group in the *syn* configuration.

Introduction. The base analogue N^4-methoxycytosine (mo⁴C) is ambivalent in its hydrogen-bonding potential, since it forms stable base pairs with both adenine and guanine in oligonucleotide duplexes (Anand, Brown & Salisbury, 1987). Watson–Crick base pairing would only be compatible with mo⁴C in the *anti* amino form, while no such constraints would be required for wobble pairing. The structure determination of the left-handed double helix