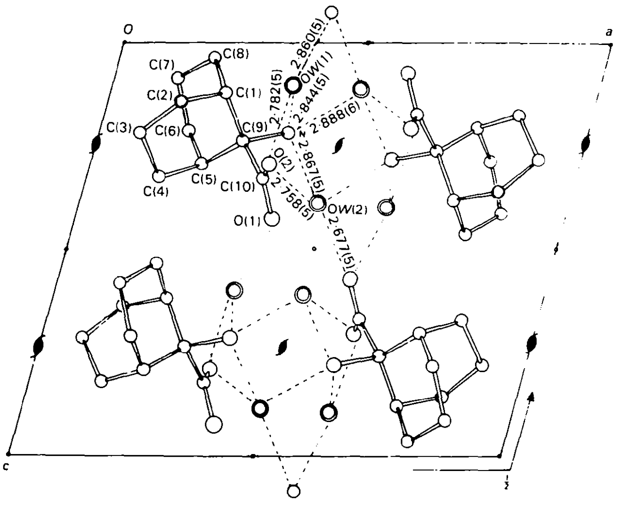

Fig. 2. Projection of the structure down the *b* axis.

The amino nitrogen (NH_3^+) forms three N—H···O hydrogen bonds with the water oxygens O*W*(1), O*W*(2) and symmetry-related O*W*(2). The two water molecules in the asymmetric unit form an extensive O—H···O hydrogen-bonded network with the symmetry-related carboxyl oxygen atoms. While O*W*(2) acts as acceptor of two N—H···O hydrogen bonds, O*W*(1) acts as acceptor of only one N—H···O hydrogen bond.

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Table 2. *Hydrogen-bonding scheme of ABN*

D—H···*A*	*D*···*A* (Å)	H···*A* (Å)	*D*—H···*A* (°)
N—H1N···O*W*(2)	2·867 (5)	1·92 (4)	152 (3)
N—H3N···O*W*(2^{ii})	2·888 (6)	1·85 (4)	175 (3)
N—H2N···O*W*(1)	2·844 (5)	1·89 (4)	150 (3)
O*W*(1)—H1*W*(1)···O(1^{iv})	2·860 (5)	1·85 (4)	179 (3)
O*W*(1)—H2*W*(1)···O(2^{i})	2·782 (5)	1·77 (4)	179 (3)
O*W*(2)—H1*W*(2)···O(2^{i})	2·758 (5)	1·94 (4)	136 (3)
O*W*(2)—H2*W*(2)···O(1^{iii})	2·677 (5)	1·79 (4)	162 (3)

Symmetry code: (i) $x, y-1, z$; (ii) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $1-x, 2-y, 1-z$; (iv) $x, \frac{3}{2}-y, z-\frac{1}{2}$.

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Structures of a Hydrocarbon $C_{58}H_{40}$ and its Photo-oxidation Products. II. 4′5′-Dihydro-1′,2,2′,3,4,4,4′-heptaphenylspiro[cyclopent-2-ene-5,5′-naphtho[2,1-*b*]-furan]-1-one, $C_{58}H_{40}O_2$

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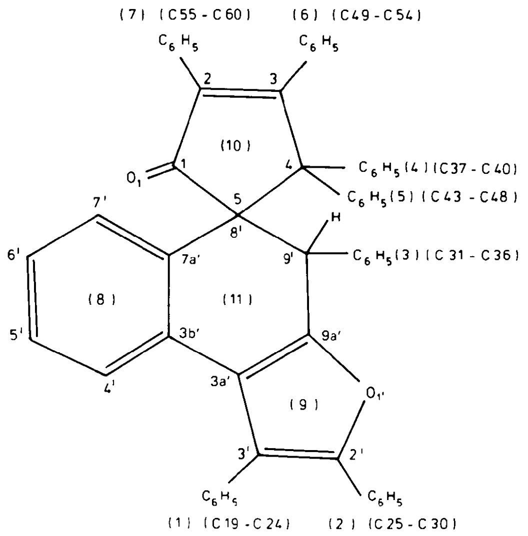
Abstract. $M_r = 768·95$, triclinic, $P\bar{1}$, $a = 12·030$ (3), $b = 13·196$ (3), $c = 13·754$ (3) Å, $\alpha = 93·27$ (2), $\beta = 100·56$ (2), $\gamma = 109·20$ (2)°, $V = 2010·9$ (8) Å^3, $Z = 2$, λ(Mo $K\alpha_1$) = 0·709300 Å, $T = 295$ (2) K, $F(000) = 808$, $D_x = 1·270$ Mg m^{-3}, $\mu = 0·070$ mm^{-1}, final $R = 0·039$ for 4171 observed data [$I > 3\sigma(I)$], 0·115 for all 7867 data. The sample was provided by M. J. Gallagher and I. D. Jenkins of the School of Chemistry, The University of New South Wales. The solution by direct methods was facilitated by the fortunate inclusion of a pair of Friedel equivalents in the reflection data. Photo-oxidation of the parent hydrocarbon was found

to have profoundly rearranged the carbon skeleton resulting in the formation of a spirocyclic system and a fused furanoid ring.

Introduction. The compound under investigation, a photo-oxidation product, is one of three compounds (Gallagher & Jenkins, 1969) whose molecular structures could not completely be determined by other means, the other two being the parent hydrocarbon $C_{58}H_{40}$ (I), and a second photo-oxidation product also of formula $C_{58}H_{40}O_2$ (III). The structures of (I) (Grainger, 1984) and (II) (herein) have now been determined by diffraction methods. Work on (III) continues. Compounds (II) and (III) are respectively the isomeric photo-oxidation products referred to as (*B*) and (*A*) in Gallagher & Jenkins (1969, p. 2613). Chemical aspects of the profound rearrangements now known to have occurred in the preparation of (I) and (II) are discussed elsewhere (Gallagher & Jenkins, 1984). A schematic drawing of (II) is shown in Fig. 1. (Note that the crystallographic numbering scheme differs from that used in the title.)

Experimental. Rhombohedral crystal, max. dimension 0·2 mm. Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ mode, $(\sin\theta)/\lambda$ to 0·62 Å^{-1}. Normal programmed e.s.d. in intensity 2%, but with 60 s time limit. Lp correction, absorption correction on 64-point grid. Cell calculated on nine reflections with $20° < \theta < 21°$. No systematic absences. 8267 reflections measured with h positive or zero, 7867 unique, $R_{\text{int}} = 0{\cdot}013$, 4171 observed $[I > 3\sigma(I)]$. Intensity statistics indicated $P\bar{1}$. Structure solved automatically on the first run by direct methods [*MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980)] without the input of structural information, giving all non-H atoms. Refinement by blocked least squares (one block each for the seven phenyl rings, one for the first six listed atoms, one for the remaining parameters). H atoms in calculated positions at fixed 1·0 Å with temperature parameters those of bonded atoms. Parameters refined: scale factor, positional and anisotropic thermal parameters for non-H atoms (541 parameters in all). Isotropic-extinction correction parameter omitted when it was found to be negligible. Refinement on $|F|$ with weights equal to reciprocal of variance of $|F|$, the variance of I being calculated as (variance based on counting statistics $+0{\cdot}0016I^2$). Final refinement, based on observed data, gave R and R_w 0·114 and 0·050 for all 7867 data, 0·039 and 0·044 for 4171 observed data; $S = 1{\cdot}0035$. Max. and r.m.s. Δ/σ in positional parameters 0·72 and 0·16. Max. and min. electron densities in final difference Fourier synthesis 0·16 and $-0{\cdot}16$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, p. 72) after adding the real part of the anomalous-dispersion correction for Mo K radiation (*International Tables*, p. 149). Principal computer programs: *MULTAN*80; *BLOCKLS*, a much modified *ORFLS* (Busing, Martin & Levy, 1962); *FANDEB* derived from *ORFFE* (Busing, Martin & Levy, 1964); and *ORTEP* (Johnson, 1976).

The original *MULTAN*80 solution ceased tangent refinement at set number 8 which had exceptionally good figures of merit. The resulting E map showed all non-hydrogen atoms. Subsequent analysis of the phases from set 8 revealed that 440 phases were correct, none were incorrect and 30 were undetermined. When refinement had been completed, and the correct phases were known, special diagnostic computer programs were employed to investigate the apparent ease with which the *MULTAN*80 programs solved such a large structure on the first attempt. The finding was that the correct phasing of reflection 062 at step 54 in the *CONVERGE* map was vital. This was achieved on the first run because the reflection data fortunately (but unintentionally) contained a Friedel pair, 031 and $0\bar{3}\bar{1}$, which *MULTAN* treated as two independent reflections 031. These were correctly phased at steps 52 and 53 and were then used to give the correct phase of 062. Without the duplicated 031 reflections fatal refinement errors were found to occur. Details of this investigation have been deposited as Appendix *A*.*



(II)

Fig. 1. Structural formula and atom labels. In the text the rings are referred to by the associated numerals (1) to (11) in parentheses.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths and bond angles, torsional angles and Appendix *A* have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39325 (39 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates of the non-hydrogen atoms* ($\times 10^4$) *and equivalent isotropic thermal parameters* B_{eq}(Å^2)

$$B_{\text{eq}} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	B_{eq}
O(1)	8699 (1)	4650 (1)	7092 (1)	3·5
C(1)	8236 (2)	3844 (2)	7464 (1)	2·7
C(2)	7256 (2)	3693 (2)	8008 (1)	2·7
C(3)	7055 (2)	2769 (2)	8424 (1)	2·6
C(4)	7933 (2)	2172 (2)	8264 (1)	2·6
C(5)	8559 (2)	2814 (2)	7400 (1)	2·6
O(1')	7663 (1)	267 (1)	5776 (1)	2·8
C(2')	8347 (2)	−398 (2)	5947 (2)	2·8
C(3')	9467 (2)	186 (2)	6495 (1)	2·6
C(3a')	9487 (2)	1278 (2)	6674 (1)	2·6
C(3b')	10377 (2)	2299 (2)	7198 (1)	2·7
C(4')	11612 (2)	2541 (2)	7311 (2)	3·3
C(5')	12413 (2)	3535 (2)	7806 (2)	3·8
C(6')	11977 (2)	4269 (2)	8210 (2)	3·5
C(7')	10746 (2)	4042 (2)	8088 (2)	3·2
C(7a')	9929 (2)	3071 (2)	7565 (1)	2·6
C(9')	7930 (2)	2185 (2)	6320 (1)	2·7
C(9a')	8385 (2)	1269 (2)	6247 (1)	2·5
C(19)	10443 (2)	−228 (2)	6900 (2)	2·7
C(20)	10660 (2)	−381 (2)	7893 (2)	4·6
C(21)	11533 (3)	−794 (2)	8279 (2)	5·5
C(22)	12201 (2)	−1076 (2)	7674 (2)	5·2
C(23)	11997 (2)	−932 (3)	6692 (2)	5·5
C(24)	11125 (2)	−510 (2)	6305 (2)	4·4
C(25)	7718 (2)	−1525 (2)	5506 (2)	3·2
C(26)	8177 (2)	−2336 (2)	5753 (2)	4·6
C(27)	7568 (3)	−3399 (2)	5308 (2)	5·4
C(28)	6493 (3)	−3663 (2)	4632 (2)	5·4
C(29)	6028 (2)	−2867 (2)	4385 (2)	5·4
C(30)	6629 (2)	−1806 (2)	4814 (2)	4·5
C(31)	8098 (2)	2859 (2)	5456 (2)	3·0
C(32)	9170 (2)	3205 (2)	5123 (2)	4·1
C(33)	9272 (3)	3791 (2)	4321 (2)	5·5
C(34)	8305 (3)	4028 (2)	3835 (2)	5·8
C(35)	7235 (3)	3695 (2)	4146 (2)	5·6
C(36)	7131 (2)	3100 (2)	4959 (2)	4·3
C(37)	8924 (2)	2401 (2)	9221 (2)	2·9
C(38)	9814 (2)	1924 (2)	9300 (2)	3·4
C(39)	10730 (2)	2159 (2)	10132 (2)	4·4
C(40)	10801 (2)	2882 (2)	10931 (2)	5·1
C(41)	9959 (3)	3382 (2)	10861 (2)	5·0
C(42)	9037 (2)	3157 (2)	10018 (2)	3·8
C(43)	7172 (2)	957 (2)	7927 (2)	2·9
C(44)	7533 (2)	96 (2)	8208 (2)	3·6
C(45)	6831 (3)	−967 (2)	7834 (2)	4·8
C(46)	5740 (3)	−1208 (2)	7192 (2)	5·3
C(47)	5328 (2)	−378 (2)	6930 (2)	4·7
C(48)	6031 (2)	688 (2)	7298 (2)	3·6
C(49)	6060 (2)	2298 (2)	8950 (2)	2·9
C(50)	6201 (2)	1784 (2)	9794 (2)	3·9
C(51)	5243 (2)	1351 (2)	10252 (2)	4·9
C(52)	4134 (2)	1396 (2)	9864 (2)	4·8
C(53)	3968 (2)	1874 (2)	9014 (2)	4·8
C(54)	4920 (2)	2328 (2)	8563 (2)	3·8
C(55)	6703 (2)	4536 (2)	8106 (2)	2·8
C(56)	6420 (2)	5056 (2)	7297 (2)	3·4
C(57)	5907 (2)	5847 (2)	7385 (2)	4·2
C(58)	5679 (2)	6133 (2)	8287 (2)	4·7
C(59)	5984 (2)	5647 (2)	9103 (2)	4·7
C(60)	6493 (2)	4857 (2)	9016 (2)	3·9

Table 2. *Bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

The e.s.d. in the last digit is 3 for distances (2 for angles) unless otherwise indicated in parentheses.

O(1)–C(1)	1·216 (2)	C(2)–C(55)	1·484	C(38)–C(39)	1·377
C(1)–C(2)	1·475	C(55)–C(56)	1·388	C(39)–C(40)	1·385 (4)
C(1)–C(5)	1·533	C(55)–C(60)	1·391	C(40)–C(41)	1·371 (4)
C(2)–C(3)	1·344	C(56)–C(57)	1·387	C(41)–C(42)	1·391
C(3)–C(4)	1·548	C(57)–C(58)	1·377	C(9')–C(31)	1·527
C(4)–C(5)	1·648	C(58)–C(59)	1·374	C(31)–C(32)	1·392
C(5)–C(7a')	1·540	C(59)–C(60)	1·382	C(31)–C(36)	1·378
C(5)–C(9')	1·582	C(3)–C(49)	1·491	C(32)–C(33)	1·383
O(1')–C(9a')	1·366 (2)	C(49)–C(50)	1·388	C(33)–C(34)	1·370 (4)
O(1')–C(2')	1·389 (2)	C(49)–C(54)	1·392	C(34)–C(35)	1·373 (4)
C(2')–C(3')	1·362	C(50)–C(51)	1·388	C(35)–C(36)	1·403 (4)
C(3')–C(3a')	1·439	C(51)–C(52)	1·366	C(2')–C(25)	1·463
C(3a')–C(3b')	1·463	C(52)–C(53)	1·373	C(25)–C(26)	1·389
C(3a')–C(9a')	1·344	C(53)–C(54)	1·380	C(25)–C(30)	1·394
C(3b')–C(7a')	1·408	C(4)–C(43)	1·555	C(26)–C(27)	1·394
C(3b')–C(4')	1·390	C(43)–C(44)	1·394	C(27)–C(28)	1·373 (4)
C(4')–C(5')	1·393	C(43)–C(48)	1·402	C(28)–C(29)	1·374 (4)
C(5')–C(6')	1·378	C(44)–C(45)	1·388	C(29)–C(30)	1·383
C(6')–C(7')	1·387	C(45)–C(46)	1·369 (4)	C(3')–C(19)	1·485
C(7')–C(7a')	1·391	C(46)–C(47)	1·383 (4)	C(19)–C(20)	1·383
C(9')–C(9a')	1·487	C(47)–C(48)	1·389	C(19)–C(24)	1·375
		C(4)–C(37)	1·545	C(20)–C(21)	1·374
		C(37)–C(38)	1·400	C(21)–C(22)	1·373 (4)
		C(37)–C(42)	1·397	C(22)–C(23)	1·363 (4)
				C(23)–C(24)	1·382

O(1)–C(1)–C(2)	125·4	C(3')–C(2')–C(25)	135·7
O(1)–C(1)–C(5)	124·7	C(2')–C(3')–C(3a')	106·2
C(2)–C(1)–C(5)	109·9	C(2')–C(3')–C(19)	127·3
C(1)–C(2)–C(3)	110·1	C(3a')–C(3')–C(19)	126·4
C(1)–C(2)–C(55)	120·9	C(3')–C(3a')–C(3b')	135·4
C(3)–C(2)–C(55)	128·8	C(3')–C(3a')–C(9a')	106·4
C(2)–C(3)–C(4)	113·5	C(3b')–C(3a')–C(9a')	118·2
C(2)–C(3)–C(49)	125·3	C(3a')–C(3b')–C(4')	123·1
C(4)–C(3)–C(49)	121·1	C(3a')–C(3b')–C(7a')	116·9
C(3)–C(4)–C(5)	101·5	C(4')–C(3b')–C(7a')	120·0
C(3)–C(4)–C(37)	109·4	C(3b')–C(4')–C(5')	120·4
C(3)–C(4)–C(43)	107·6	C(4')–C(5')–C(6')	119·7
C(37)–C(4)–C(43)	115·0	C(5')–C(6')–C(7')	120·4
C(5)–C(4)–C(37)	107·9	C(6')–C(7')–C(7a')	120·8
C(5)–C(4)–C(43)	114·6	C(3b')–C(7a')–C(5)	119·7
C(1)–C(5)–C(4)	101·8 (1)	C(3b')–C(7a')–C(7')	118·6
C(1)–C(5)–C(7a')	112·0	C(5)–C(7a')–C(7')	121·7
C(1)–C(5)–C(9')	106·9	C(5)–C(9')–C(9a')	105·2
C(4)–C(5)–C(7a')	114·5	C(5)–C(9')–C(31)	116·0
C(4)–C(5)–C(9')	111·7	C(9a')–C(9')–C(31)	112·8
C(7a')–C(5)–C(9')	109·4	C(3a')–C(9a')–C(9')	126·3
C(2')–O(1')–C(9a')	105·9	O(1')–C(9a')–C(9')	122·0
O(1')–C(2')–C(3')	110·0	C(3a')–C(9a')–O(1')	111·5
O(1')–C(2')–C(25)	114·3		

C(1)–C(2)–C(3)–C(4)	3·0	C(3a')–C(3b')–C(7a')–C(5)	−0·1 (3)
C(2)–C(3)–C(4)–C(5)	−12·9	C(3b')–C(7a')–C(5)–C(9')	−39·2
C(3)–C(4)–C(5)–C(1)	16·8	C(7a')–C(5)–C(9')–C(9a')	51·8
C(4)–C(5)–C(1)–C(2)	−16·5	C(5)–C(9')–C(9a')–C(3a')	−33·6 (3)
C(5)–C(1)–C(2)–C(3)	9·5	C(9')–C(9a')–C(3a')–C(3b')	−5·4 (3)
O(1)–C(1)–C(2)–C(3)	−172·6	C(9a')–C(3a')–C(3b')–C(7a')	24·6 (3)
C(2)–C(3)–C(4)–C(37)	100·8		
C(2)–C(3)–C(4)–C(43)	−133·6		

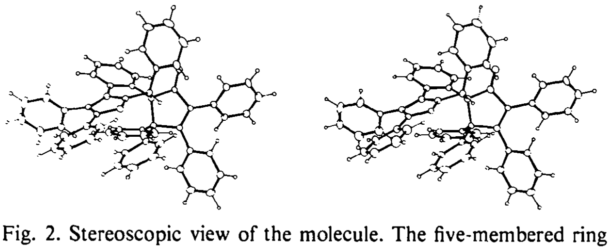

Fig. 2. Stereoscopic view of the molecule. The five-membered ring (10) is situated near the centre of the diagram parallel to the page, and ring (9) is a little to the left. Ring (10) is attached to (11) *via* the spiro atom C(5) which is partly obscured by C(9'). O(1) is partly obscured by C(36). Proceeding clockwise from the uppermost ring, the six-membered rings are numbers (3), (7), (6), (5), (4), (2), (1) and (8).

Discussion. The stereoscopic view of the molecule in Fig. 2 shows it to comprise mainly near-planar rings. Table 1 lists atomic coordinates, and Table 2 selected bond lengths and angles.

Within the phenyl rings numbered (as in Fig. 1) (1), (2), (3), (4), (5), (6) and (7), the benzo ring (8), and the five-membered ring (9), the largest deviations from the least-squares planes are only 0·004, 0·006, 0·004, 0·016, 0·022, 0·012, 0·013, 0·022 and 0·011 Å (e.s.d. = 0·003 Å) respectively. Ring (10) is somewhat more distorted, the deviations of atoms C(1) to C(5) from the plane being 0·090, −0·021, −0·057, 0·099 and −0·111 Å (σ = 0·002 Å) respectively, atom O(1) being 0·272 (1) Å from the plane. Ring (11) is highly distorted with deviations from the plane as great as 0·33 and −0·31 Å for C(5) and C(9′) respectively, and torsional angles as large as 51·8 (2)° (Table 2). The angles between the normals to the pairs of planes (8) and (9), (8) and (11), (9) and (11) are 27·3, 18·3 and 20·3° (σ = 0·3°) respectively. Thermal anisotropy is moderate in the central rings but significant in some of the attached phenyl rings. There is a marked tendency for the various planar sections of the molecule to be parallel to one another and/or to some significant repeat direction such as a cell axis. All intermolecular contacts are above van der Waals distances.

Thanks are due to Professor M. J. Gallagher (School of Chemistry, The University of New South Wales) for suggesting the problem, for providing crystals and for helpful discussions, to Mr D. C. Craig for data collection and assistance in computing, to Mrs C. Faust for drawing the diagrams computed by *ORTEP* (Johnson, 1976), and to the University for computing time on the CDC-171. Bond lengths and angles were computed using *FANDEB*, a local version of *ORFFE* (Busing, Martin & Levy, 1964).

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Thiocarbonohydrazide Dihydrochloride Hemihydrate, $CH_8N_4S^{2+}.2Cl^-.0{\cdot}5H_2O$: A Triclinic Structure which Exhibits a Monoclinic Subcell

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Abstract. M_r = 188·08, triclinic, $P\overline{1}$, a = 9·936 (7), b = 19·108 (9), c = 9·421 (6) Å, α = 104·5 (1), β = 118·4 (1), γ = 90·9 (1)°, V = 1504 (2) Å^3, Z = 8, D_x = 1·66 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 10·58 cm^{-1}, $F(000)$ = 776, T = 295 K, R = 0·0322 for 3884 independent observed reflections. There are four crystallographically independent units in the cell, approximately related by pseudo symmetry centres and twofold screw axes, so a monoclinic subcell can be recognized. The structure consists of diprotonated cations, in the expected *cis,cis* conformation, of Cl^- anions and of water molecules. The structural parameters in the four independent units are practically equal. Several $>NH\cdots Cl$, $-NH_3^+\cdots Cl$, $-NH_3^+\cdots O$ and $OH\cdots Cl$ hydrogen bonds determine the packing of the cations and anions in the crystal.

Introduction. Thiocarbonohydrazide (tcaz), $SC(NHNH_2)_2$, and its protonated species can assume either a *cis,trans* conformation, *i.e.* with one hydrazinic group bent toward the C—S bond and the other toward the opposite side, or a *cis,cis* conformation, *i.e.* with both groups bent toward the C—S bond. The former has been found in the neutral molecule (Braibanti, Tiripicchio & Tiripicchio Camellini, 1969, 1972), in its metal chelates (*N,S*) (Bigoli, Braibanti, Manotti Lanfredi, Tiripicchio & Tiripicchio Camellini, 1971; Bigoli, Pellinghelli, Tiripicchio & Tiripicchio Camellini, 1975; Bigoli, Pellinghelli & Tiripicchio, 1975) and when tcaz is monoprotonated (Braibanti, Tiripicchio & Tiripicchio Camellini, 1972); the latter is assumed when tcaz is diprotonated (Braibanti, Pellinghelli, Tiripicchio & Tiripicchio Camellini, 1971; Bigoli, Braibanti, Manotti

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