the molecule prepared using *ORTEP*II (Johnson 1976).

Discussion. The analysis shows that the dithiin ring adopts a half-chair conformation. Atoms C5, C6, S1 and S4 define a plane (plane 1; maximum deviation 0·004 Å) with C2 and C3 0·519 (4) and −0·351 (4) Å from the plane, respectively. The phenyl-ring plane is almost normal (interplanar angle 93·2°) to plane 1. Relief from steric strain to which the *cis* H—C6═C5—C11 configuration might have given rise is achieved by significant bond angle distortion {C11—C5—C6 is 124·2 (3)°, greater than C11—C5—S4 [110·4 (2)°]} and by out-of-plane bending of C5 from the phenyl-ring plane [C5 is 0·121 (4) Å off the phenyl plane in a direction consistent with the phenyl plane being bent away from the hydrogen at C6].

The C5═C6 double bond [1·321 (5) Å] does not vary significantly from the corresponding values in 2,3-dihydro-5,6-dimethyl-1,4-dithiin 1,1,4,4-tetroxide (II) [1·320 (4) Å] and 2,3-dihydro-5-methyl-1,4-dithiin 1,1,4,4-tetroxide (III) [1·338 (8) Å]. The S—C(sp^2) bond lengths [1·764 (3) and 1·779 (3) Å] are comparable with the corresponding distances in (II) [mean 1·779 (4) Å] and in (III) [mean 1·762 (6) Å]. The S—C(sp^3) distances [1·767 (3), 1·768 (3) Å] are also similar to those in (II) [mean 1·766 (3) Å] and (III) [mean 1·787 (7) Å]. The value quoted (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987) for such bonds is 1·779 (20) Å for 94 samples, and with lower and upper quartile values of 1·764 and 1·790 Å respectively.

The average distance of an S═O bond in the molecule [1·435 (3) Å] is very close to the value listed [1·436 (10) Å from 316 samples] for bonds of this type (Allen *et al.*, 1987). The CH_2—CH_2 bond length [C2—C3 1·503 (5) Å] and other dimensions are also unexceptional; there are no untoward intermolecular contacts, the shortest being O11···C6 (at $-\frac{1}{2}-x$, $1-y$, $-\frac{1}{2}+z$) [3·199 (4) Å].

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Structure of 1,3-Cyclopentanedione

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Abstract. 1,3-Cyclopentanedione, $C_5H_6O_2$, M_r = 98·10, monoclinic, $C2/c$, a = 7·451 (2), b = 12·853 (3), c = 10·754 (2) Å, β = 111·90 (2)°, V = 956·2 (3) Å^3, Z = 8, D_x = 1·37 g cm^{-3}, m.p. 422–425 K, λ(Mo $K\alpha$) = 0·71069 Å, μ = 0·65 cm^{-1}, $F(000)$ = 416, T = 292 K, final R = 0·083 for 486 observed reflections. The molecules adopt the enol form and are connected head-to-tail by hydrogen bonds in

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which the O(1)···O(2′) distance is 2·542 (8) Å. The molecules lie in chains approximately in one plane parallel to **b** and inclined by about 12° to the crystallographic plane (100); the molecules themselves are planar, within error limits. Strong elongation of the vibrational ellipsoids of non-H atoms perpendicular to the plane of the molecules is observed. A pressure-generated phase transition was observed in the crystals of 1,3-cyclopentanedione below 0·3 GPa.

Introduction. This X-ray structural determination of 1,3-cyclopentanedione (CPD) continues our studies on the structures of simple cyclic β-diketoalkanes. Most recently we reported the structure of 2-methyl-1,3-cyclopentanedione (Katrusiak, 1989). In the crystalline state cyclic β-diketoalkanes are present in the enol form and they form aggregates of the molecules interconnected by strong hydrogen bonds (shorter than 2·6 Å) between their hydroxyl and carbonyl oxygens. Several stereochemical configurations of the hydrogen bonds are possible according to the position of the hydrogen with respect to the bond to C(2) (Etter, Urbańczyk-Lipkowska, Jahn & Frye, 1986). The following configurations of hydrogen bonds were found in the structures of cyclic β-diketoalkanes (the hydroxyl orientation is specified first and the carbonyl hydrogen-bond orientation second): the *anti-anti* configuration in 1,3-cyclohexanedione (CHD), *syn-anti* configuration in the 6:1 CHD:benzene inclusion cystals (6CHD:B) (Etter, Urbańczyk-Lipkowska, Jahn & Frye, 1986), *anti-syn* in 2-methyl-1,3-cyclopentanedione (MCPD) and *syn-syn* in dimedone (*i.e.* 5,5-dimethyl-1,3-cyclohexanedione) (Semmingsen, 1974; Singh & Calvo, 1975).

In the present study on CPD we were particularly interested in the configuration and geometry of the hydrogen bond, in the dimensions of the HO—C=C—C=O fragment of the CPD molecule and in the arrangement of the molecules in the crystal lattice. We also intended to investigate the structure of the CPD crystals at high pressure, together with the high-pressure structure determinations carried out for the crystals of MCPD, CHD and dimedone (Katrusiak, 1990). All the high-pressure determinations in this series were concerned with complex studies of intermolecular interactions in molecular crystals (hydrogen bonds in particular), of possible phase transitions in CPD and CHD and of the changes in the structure of the molecules themselves at high pressures, the latter subject being the continuation of our previous studies on the influence of intermolecular forces on conformational changes of molecules in organic crystals made by comparing symmetry-independent molecules (Katrusiak & Kałuski, 1980). Unfortunately, the sample crystals of CPD were damaged when the pressure was elevated to about 0·1–0·3 GPa, this being most certainly caused by a phase transition in the CPD crystals in that pressure range. This phase transition could be observed by eye in a high-pressure diamond-anvil cell as it was marked, apart from the sample-crystal breakage at high pressure, by a significant change in the shape of the small crystal fragments. Several sample crystals were mounted in the high-pressure cell and several attempts were made to elevate the pressure to about 0·5 GPa, all of them unsuccessful. In the similar structural investigation of CHD, a phase transition was found which takes place over the pressure range 0·1 MPa to 0·125 GPa (Katrusiak, 1990); this phase transition strongly deformed the (ambient-pressure) unit cell of CHD and changed the shape of the sample crystal so noticeably that the phase transition could be clearly observed by eye. The CHD crystals, unlike the CPD samples, were not damaged and could be brought through the phase transition many times without any deterioration in quality. We expect that the crystals of CPD and CHD undergo a similar pressure-driven phase transition, because they are very similar in that in both these structures the molecules are interconnected by *anti-anti* hydrogen bonds and form chains of molecules lying approximately in one plane. The detailed description of the phase transitions in CHD and the experimental procedure of our high-pressure investigation of CPD have been published elsewhere (Katrusiak, 1990).

Experimental. We found it very difficult to obtain high quality CPD crystals suitable for X-ray diffraction experiments; CPD is unstable and should be kept in dry and cold conditions. Finally, small crystals (elongated plates) resublimed in the test-tube walls above the ethanol solution of CPD. They were slightly yellow which indicated that some of the substance underwent decomposition and many of the crystals were twinned; the crystals seemed stable and no capillary was used. The unit-cell parameters and space group were determined by the precession method, the crystal faces were parallel to crystallographic planes (001), (010) and ($\bar{2}01$). The sample crystal selected for data collection had dimensions 0·2 × 0·2 × 0·2 mm and was a fragment cut from a longer crystal. A CAD-4 diffractometer and graphite-monochromated Mo $K\alpha$ radiation were used. The orientation matrix was based on 16 automatically centred reflections with $8{\cdot}0 < 2\theta < 24{\cdot}0°$. The intensities were measured with a stationary background and 2θ–θ scan mode with a variable scan speed (1·0–20·0° min^{-1}) depending on reflection intensity. 955 reflections were measured out to $2\theta = 50°$ (h 0→9, k 0→16, l −13→13), 754 of them independent ($R_{\rm merge} = 0{\cdot}014$) and 440 with $I \leq 2{\cdot}25\sigma_I$. Two control reflections monitored every two

Table 1. *Coordinates and equivalent isotropic thermal parameters* ($\AA^2$) *for non*-H *atoms and coordinates and* $U_{\rm iso}$ *for* H(1)

$U_{\rm eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U
O(1)	0·2219 (9)	0·4481 (3)	0·1399 (4)	0·070
O(2)	0·2308 (9)	0·8096 (3)	0·1820 (5)	0·079
C(1)	0·2363 (12)	0·5415 (5)	0·1913 (7)	0·060
C(2)	0·2162 (11)	0·6309 (4)	0·1229 (6)	0·046
C(3)	0·2437 (11)	0·7159 (5)	0·2126 (6)	0·053
C(4)	0·2838 (13)	0·6759 (5)	0·3508 (7)	0·055
C(5)	0·2805 (12)	0·5569 (5)	0·3364 (6)	0·051
H(1)	0·311 (14)	0·393 (6)	0·246 (10)	0·14 (3)

hours showed no systematic variation in intensity throughout the data collection. Lp corrections were applied, no absorption correction.

The structure was solved by direct methods (Sheldrick, 1986); the non-H atoms were refined anisotropically and the H atoms were found from a ΔF map; they were included in the refinement with isotropic temperature factors but they refined to unrealistic positions and were substituted by H atoms calculated from the geometry of the molecule, their coordinates being recalculated after each cycle of refinement; only H(1) was free to refine. Seven strong reflections with $\Delta F/\sigma > 5{\cdot}0$ were excluded from the final cycles of refinement. The function $\sum w(|F_o| - |F_c|)^2$ was minimized where $w = \sigma_{F_o}^{-2}$. The final $R = 0{\cdot}083$, $wR = 0{\cdot}083$, $S = 1{\cdot}9$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}008$ and the highest and the lowest densities in the final ΔF map were 0·26 and $-0{\cdot}30\ {\rm e\ \AA^{-3}}$, respectively. The atomic coordinates for the refined atoms, $U_{\rm eq}$ for non-H atoms and $U_{\rm iso}$ for H(1) are listed in Table 1.* Most of the calculations were performed with *SHELX76* (Sheldrick, 1976) on an IBM XT Turbo computer, atomic scattering factors were those incorporated in *SHELX76*.

Discussion. The molecule of CPD and its atomic labelling is presented in Fig. 1. The anisotropic thermal parameters indicate strong elongation of the ellipsoids of thermal vibrations along x, *i.e.* perpendicular to the plane of the ring; the values of $U(11)$ are the largest for all non-H atoms and for the two O atoms they are even larger than 0·1 $\AA^2$. The reasons for these large values could be real vibrations of the molecules in the crystal lattice, disorder or errors due to the defects in the crystal sample. One or some of these effects could cause inconsistencies between the real structure and its model presented here, as demonstrated by the high R values and relatively high e.s.d.'s of the refined parameters. The uncertain nature of these high vibrational parameters restrained us from applying any corrections to the geometry of the molecule. The bond lengths and valency angles are listed in Table 2, which also contains the corresponding values observed in MCPD, for comparison. The bond orders corresponding to the lengths of the bonds HO(1)—C(1)=C(2)—C(3)=O(2) are 1·4, 2·0, 1·3 and 1·8 Å, respectively (Allmann, 1977); as observed in the previously investigated structures of MCPD, CHD or dimedone, these bonds are well defined in this alternate π-electron system. The lengths of all bonds in CPD, except C(1)—C(2), agree (*i.e.* differ by less than three e.s.d.'s) with those in MCPD; the bond C(1)=C(2) is significantly shorter in CPD than the corresponding bond in MCPD, CHD or dimedone, and the bond order of C(1)=C(2) in CPD also seems too high; hence we suppose that this apparent shortening of C(1)—C(2) could be due to the errors discussed above. The bonds C(1)—C(5) and C(3)—C(4) are both slightly shorter in CPD than in MCPD, but agree very well with the corresponding bonds observed in CHD, 6CHD:B and dimedone. Angular distortions within the five-membered ring (from a geometrically ideal value of 103°) are very similar in CPD and MCPD; the greatest difference between these structures is 1·4 (6)° (*i.e.* 2·3 e.s.d.'s) [observed for the angle C(1)=C(2)—C(3)] and can be attributed to the influence of the methyl substituent at C(2) in MCPD. Valency angles HO(1)—C(1)=C(2) and C(2)—C(3)=O(2), external to the pentanedione ring, have the same values within error, while they are significantly different in MCPD. The CPD molecule is planar, although its planarity is not restricted by the crystallographic symmetry (as it was in MCPD): $\chi^2 = 2{\cdot}64$ for the least-squares plane fitted to all non-H atoms.

The arrangement of the CPD molecules in the crystal lattice is shown in two projections in Fig. 2. The molecules are interconnected by hydrogen bonds and form chains along **b**; the molecules within one

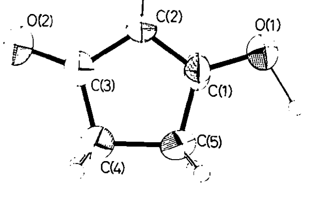

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the 1,3-cyclopentanedione molecule viewed perpendicular to the plane of its ring; the ellipsoids are drawn at the 50% probability level, H atoms are represented as small circles.

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* Lists of anisotropic thermal parameters, least-squares-plane data, H-atom positions and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52691 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and valency angles* (°) *in* CPD; *the corresponding values in* MCPD (*Katrusiak*, 1989) *are also given for comparison*

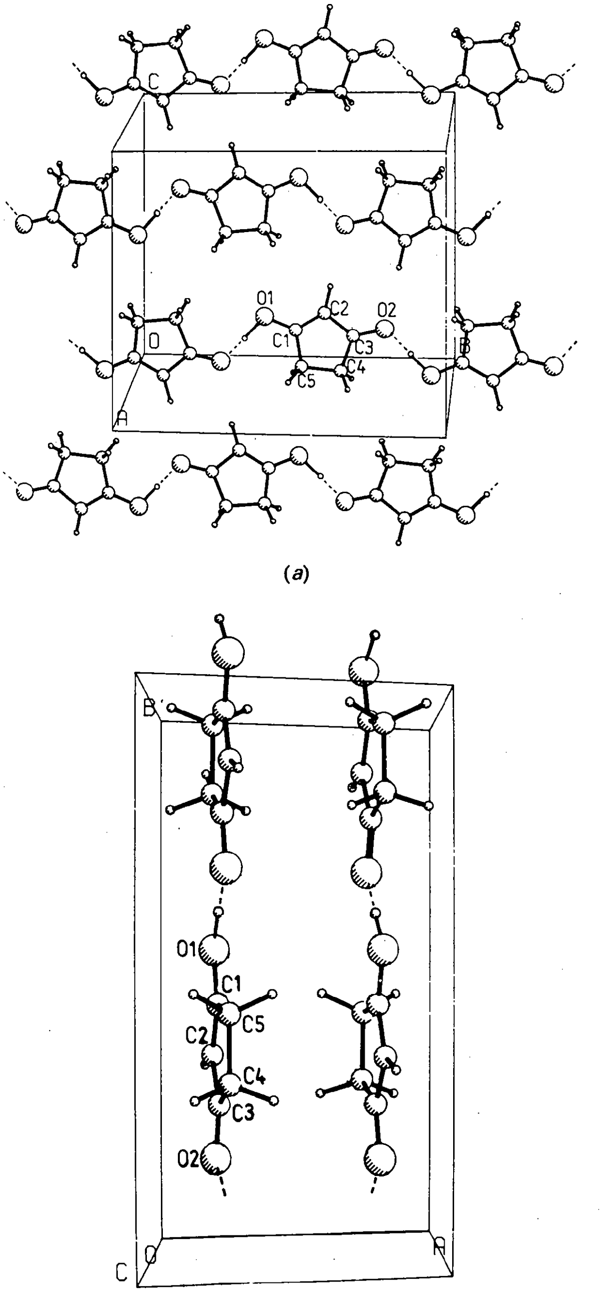
	CPD	MCPD		CPD	MCPD
O(1)—C(1)	1·309 (7)	1·328 (2)	C(1)—C(5)	1·484 (8)	1·502 (3)
O(2)—C(3)	1·243 (7)	1·235 (2)	C(3)—C(4)	1·495 (9)	1·508 (3)
C(1)—C(2)	1·304 (8)	1·358 (3)	C(4)—C(5)	1·536 (8)	1·525 (3)
C(2)—C(3)	1·421 (8)	1·429 (3)			

	CPD	MCPD
O(1)—C(1)—C(2)	125·4 (6)	123·96 (15)
O(2)—C(3)—C(2)	126·0 (6)	127·30 (17)
O(1)—C(1)—C(5)	121·2 (6)	121·96 (15)
O(2)—C(3)—C(4)	124·3 (6)	122·34 (16)
C(2)—C(1)—C(5)	113·4 (6)	114·07 (15)
C(2)—C(3)—C(4)	109·7 (5)	110·37 (15)
C(1)—C(5)—C(4)	103·2 (5)	102·99 (14)
C(3)—C(4)—C(5)	104·6 (5)	104·90 (15)
C(1)—C(2)—C(3)	109·1 (6)	107·67 (15)

chain lie, within error, in one plane which is parallel to **b** and at an angle of about 12° to the crystallographic plane (100). The O(1)···O(2) distance in this hydrogen bond is 2·542 (8) Å.

In view of the low accuracy in the position of H(1) its location in the hydrogen bond will not be discussed here. The hydrogen bond in CPD is the shortest among the cyclic β-diketoalkanes investigated, but the differences in lengths are very small or even statistically insignificant: the length of the *anti-anti* hydrogen bond in CHD was 2·561 (4) Å, the *syn-anti* hydrogen bond in 6CHD:B was 2·579 (1) Å, the *syn-syn* hydrogen bond in dimedone was 2·595 (2) Å and the *anti-syn* hydrogen bond in MCPD was 2·598 (2) Å; two independent hydrogen bonds observed in squaric acid (3,4-dihydroxy-3-cyclobutene-1,2-dione) were 2·553 (1) and 2·554 (1) Å (Semmingsen, Hollander & Koetzle, 1977).

The arrangement of the CPD molecules within one chain is similar to that observed in CHD in that the molecules lie approximately in one plane and that the neighbouring molecules in the chain assume an opposite orientation: in CPD the neighbouring molecules are related by a twofold screw axis, while in CHD the neighbouring molecules (chains are along the crystallographic [201] direction for the unit cell of symmetry $P2_1/c$) can be transformed one into another by an operation combining a glide *c* and a translation (1,0,0). One molecule of CHD occupies a slightly longer interval in the chain [7·086 (5) Å] than one CPD molecule [6·426 (3) Å, *i.e.* *b*/2]; a very similar interval was taken up by one molecule in the MCPD crystal [6·4814 (8) Å, *i.e.* *a*, since the neighbouring molecules of MCPD are related by a translation (1,0,0)]. The CPD molecules are inclined to the central line of the chain (collinear with the twofold screw axis) by an angle $\rho = 5{\cdot}32\,(1)^\circ$ [the angle ρ is defined as the angle between the line drawn through O(1) and O(2) and the central line of the chain] in a way which minimizes the electrostatic interactions of the molecules in one chain. A similar orientation of the molecules, with the carbonyl O atom closer to the central line of the chain than the hydroxyl O



(*a*)

(*b*)

Fig. 2. Arrangement of the 1,3-cyclopentanedione molecules in the crystal lattice (Motherwell, 1976): (*a*) a perspective view of the chains of the hydrogen-bonded molecules along **a**; (*b*) a perspective view of the chains down **c**. In both these drawings only half of the unit-cell contents have been plotted to avoid overlapping of the molecules.

atom, was also observed in the ambient-pressure and high-pressure structures of CHD (Katrusiak, 1990).

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N-Methyl-*N'*-sulfamidoisothiourea

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Abstract. (1,2-Dimethyl-3-isothiourea)sulfonamide, $C_3H_9N_3O_2S_2$, $M_r = 183{\cdot}3$, triclinic, $P\bar{1}$, $a = 7{\cdot}216\,(2)$, $b = 7{\cdot}932\,(3)$, $c = 8{\cdot}201\,(2)$ Å, $\alpha = 66{\cdot}37\,(2)$, $\beta = 84{\cdot}78\,(2)$, $\gamma = 64{\cdot}10\,(2)^\circ$, $V = 385$ Å^3, $Z = 2$, $D_x = 1{\cdot}58$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}54178$ Å, $\mu = 5{\cdot}82$ mm^{-1}, $F(000) = 192$, room temperature, $R = 0{\cdot}041$ for 1026 observed reflections with $|F_o| > 3\sigma(|F_o|)$. The C—S bond to the methylated sulfur is longer than the analogous bond in urea. The C—N and N—S bonds show considerable delocalization. An intramolecular hydrogen bond exists between the methylated amine hydrogen and an oxygen of the sulfamide group.

Introduction. We are interested in cyclization reactions at metal centres and have prepared several unusual metal–sulfur–nitrogen complexes (Kelly & Woollins, 1986; Jones, Kelly, Williams & Woollins, 1988; Jones, Kelly, Warrens, Williams & Woollins, 1986). Recently, there has been a report on sulfamido–isothiourea derivatives (Michael, Ross & Rees, 1985) which have been used in *organic* cyclization reactions. The potential for these compounds in *coordination* chemistry has not been investigated and structural information is not available. We are involved in a study of the coordinating properties of this type of compound with a view to forming new materials with interesting physical properties. Prior to a study of their complexing ability we have determined the structure of one example, *N*-methyl-*N'*-sulfamidoisothiourea (1).

Experimental. Colourless single crystals of (1) suitable for X-ray crystallography were grown at room temperature by slow diffusion of *n*-hexane into a dichloromethane solution of the compound. Crystal size 0·16 × 0·14 × 0·20 mm. Refined unit-cell parameters obtained from setting angles of 16 reflections with $11{\cdot}8 \le \theta \le 28{\cdot}1^\circ$. Nicolet *R*3*m* diffractometer. 1039 independent reflections ($2\theta \le 116^\circ$), Cu $K\alpha$ radiation (graphite monochromator), ω scan. 1026 $[|F_o| > 3\sigma(|F_o|)]$ considered observed, index range h −7/7, k −7/8, l 0/8; two check reflections measured every 50 reflections, no significant change in net counts; Lorentz and polarization corrections, numerical absorption correction (face-indexed crystal), maximum and minimum transmission factors 0·460 and 0·230. Structure solved by direct methods. The non-H atoms were refined anisotropically. The leading protons on the methyl groups were located from a ΔF map. The protons on N(1) and N(3) were located from a ΔF map and refined isotropically. The positions of the remaining H atoms were idealized C—H 0·96 Å, assigned isotropic thermal parameters,

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