found to be the same in both studies, as revealed by comparing least-squares-plane analyses and dihedral angles. With the exception of the angle C(1)—N(1)—C(15)—N(2), all dihedral angles are in excellent agreement (an average difference of 1·5 e.s.d.'s). Finally, the distance between the N and O atoms in the N—H···O hydrogen bond is identical in both structures.

The authors in addition wish to recognize the work of Chang, Yang, Yoo, Wang, Pletcher & Sax (1981) on the crystal structure determination of carbamazepine acetone solvate.

References

CARPY, P. A., GADRET, M., GOURSOLLE, M., LEGER, J. M. & LEHUEDE, J. (1979). *Acta Cryst.* B**35**, 996–998.

CHANG, C.-H., YANG, D. S. C., YOO, C. S., WANG, B.-C., PLETCHER, J. & SAX, M. (1981). *Acta Cryst.* A**37**, C71.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

DE CAMP, W. H., BRANNON, W. L. & MAIENTHAL, M. M. (1981). In preparation.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

HOEK, W. G. M. VAN DEN, OONK, H. A. J. & KROON, J. (1979). *Acta Cryst.* B**35**, 1858–1861.

International Tables for X-ray Crystallography (1969). Vol. I, 3rd ed., pp. 530–535. Birmingham: Kynoch Press.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

KOJIĆ-PRODIĆ, B., RUŽIĆ-TOROŠ, Ž. & GOLIČ, L. (1980). *Acta Cryst.* B**36**, 388–392.

LINDNER, H. J. & VON GROSS, B. (1972). *Chem. Ber.* **105**, 434–440.

PAUL, I. C., JOHNSON, S. M., PAQUETTE, L. A., BARRETT, J. H. & HALUSKA, R. J. (1968). *J. Am. Chem. Soc.* **90**, 5023–5024.

REBOUL, J. P., CRISTAU, B., SOYFER, J. C. & ASTIER, J. P. (1981). *Acta Cryst.* B**37**, 1844–1848.

REBOUL, J. P., CRISTAU, B., SOYFER, J. C. & ESTIENNE, J. (1980). *Acta Cryst.* B**36**, 2683–2688.

STEWART, J. M., MACHIN, P. A., DICKINSON, C., AMMON, H. L., HECK, H. & FLACK, H. (1976). The XRAY system – version of March 1976. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1981). B**37**, 2245–2248

The Structure of the Orthorhombic Form of Tolbutamide (1-*n*-Butyl-3-*p*-toluenesulphonylurea)

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Abstract. $C_{12}H_{18}N_2O_3S$ (form *A*), orthorhombic, $Pna2_1$, $a = 20{\cdot}223\,(11)$, $b = 7{\cdot}831\,(9)$, $c = 9{\cdot}090\,(10)$ Å, $Z = 4$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $D_x = 1{\cdot}246$, D_m (flotation) = $1{\cdot}251$ Mg m^{-3}. $R = 0{\cdot}048$ for 988 reflections. The four molecules in the unit cell occur in hydrogen-bonded pairs.

Introduction. Many drugs exist in two or more polymorphic forms, and in some cases such polymorphs have been shown to have different levels of bio-availability, *e.g.* cortisone acetate (Macek, 1954). On the other hand, the polymorphs of acetylhexamide show no such differences (Muller & Lagas, 1979). The current elucidation of the crystal structure of tolbutamide was undertaken to study the differences between the polymorphs of this compound which might lead to one or other type of behaviour. Two polymorphs of tolbutamide are known (Simmons, Ranz, Gyanchandani & Picotte, 1972; Leary, Ross & Thomas, 1981), and the orthorhombic form, designated as form *A* by Simmons, is the form normally used in pharmaceutical preparations. This material was first synthesized by Makhnenko & Sysoeva (1959) and crystallizes as plates on the addition of *n*-hexane to a

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solution in benzene. Systematic absences were found to be *h*0*l* with *h* odd, 0*kl* with *k* + *l* odd, and 00*l* with *l* odd, consistent with the space groups *Pnam* or $Pna2_1$. Since $Z = 4$ and only $Pna2_1$ has fourfold general positions, this must be the correct space group. The other polymorph, Simmons's form *B*, has been shown to be monoclinic with $Z = 2$ (Leary, Ross & Thomas, 1981) and crystallizes as fibrous needles from ethanolic solution on addition of water. Elemental analysis of form *A* prepared by Makhnenko's method gave C 53·42 (53·31); H 6·75 (6·71); N 10·27 (10·36); S 11·76% (11·86%). The values in parentheses are the theoretical percentages for $C_{12}H_{18}N_2O_3S$.

Intensities were collected on an Enraf–Nonius CAD-4F *κ*-geometry diffractometer with graphite-monochromated Mo *Kα* radiation at the Polytechnic of North London. 1356 independent reflections were recorded. Lp corrections were applied and the data output as condensed data cards with *SHELX*. The positions of the atoms in the SO_2 group were found with *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); the other atoms were not indicated definitely in this way. The remaining atoms, other than H, were located from difference syntheses; least-squares refinement led to $R = 0{\cdot}13$. The H atoms were then added geometrically and *R* dropped to 0·083. Refinement with anisotropic temperature factors for atoms other than H reduced *R* to 0·068. At this stage, 366 reflections for which $I < 2{\cdot}5\sigma(I)$, and 2 reflections for which $F_c < 0{\cdot}5F_o$ were omitted, leaving 988 reflections. The final *R* was 0·048 with unit weights. All computations were carried out on the CDC 7600 computer at the University of London computer centre with *MULTAN* 78, *SHELX* 76 and *LSHELX* 76 (Sheldrick, 1976).

Table 1. *Fractional atomic positions and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$U_{eq} = \frac{1}{3}\,\text{trace}\,\tilde{\mathbf{U}}$.

	x	*y*	*z*	U_{eq} (Å^2)
S(1)	0·8962 (1)	0·1636 (2)	0·3922 (0)	0·0466 (8)
O(2)	0·9043 (2)	0·2543 (7)	0·2555 (5)	0·056 (5)
O(3)	0·8670 (2)	0·2502 (7)	0·5148 (5)	0·059 (3)
O(4)	1·0021 (3)	−0·0293 (7)	0·2417 (5)	0·060 (3)
C(5)	1·0073 (3)	−0·0169 (9)	0·3796 (8)	0·051 (4)
C(6)	1·0853 (3)	−0·2534 (10)	0·3967 (12)	0·065 (5)
H(7)	1·1044 (3)	−0·2132 (10)	0·2913 (10)	
H(8)	1·1260 (3)	−0·2841 (10)	0·4690 (10)	
C(9)	1·0443 (5)	−0·4143 (11)	0·3766 (15)	0·095 (7)
H(10)	1·0268 (5)	−0·4565 (11)	0·4829 (15)	
H(11)	1·0025 (5)	−0·3815 (11)	0·3081 (15)	
C(12)	1·0811 (6)	−0·5627 (14)	0·3004 (16)	0·119 (10)
H(13)	1·1082 (6)	−0·5180 (14)	0·2056 (16)	
H(14)	1·0473 (6)	−0·6623 (14)	0·2672 (16)	
C(15)	1·1254 (7)	−0·6358 (16)	0·4093 (30)	0·191 (16)
H(16)	1·1568 (7)	−0·7142 (16)	0·3410 (30)	
H(17)	1·1133 (7)	−0·7042 (16)	0·5089 (30)	
H(18)	1·1509 (7)	−0·5191 (16)	0·4370 (30)	
C(19)	0·7465 (5)	−0·4907 (12)	0·2638 (14)	0·097 (7)
H(20)	0·7370 (5)	−0·5758 (12)	0·3548 (14)	
H(21)	0·7822 (5)	−0·5481 (12)	0·1907 (14)	
H(22)	0·7010 (5)	−0·4677 (12)	0·2046 (14)	
N(23)	0·9685 (3)	0·0999 (8)	0·4568 (6)	0·046 (3)
H(24)	0·975 (4)	0·105 (11)	0·560 (10)	
N(25)	1·0473 (3)	−0·1120 (8)	0·4607 (6)	0·049 (3)
H(26)	1·050 (4)	−0·076 (12)	0·584 (11)	
C(27)	0·8525 (3)	−0·0267 (10)	0·3573 (7)	0·048 (4)
C(28)	0·8589 (4)	−0·1643 (10)	0·4518 (9)	0·062 (5)
H(29)	0·8908 (4)	−0·1534 (10)	0·5467 (9)	
C(30)	0·8243 (4)	−0·3126 (10)	0·4254 (10)	0·069 (5)
H(31)	0·8293 (4)	−0·4200 (10)	0·4991 (10)	
C(32)	0·7825 (4)	−0·3251 (11)	0·2993 (10)	0·067 (6)
C(33)	0·7760 (4)	−0·1831 (12)	0·2093 (10)	0·077 (6)
H(34)	0·7429 (4)	−0·1903 (12)	0·1162 (10)	
C(35)	0·8111 (4)	−0·0342 (11)	0·2346 (10)	0·067 (5)
H(36)	0·8057 (4)	0·0755 (11)	0·1636 (10)	

Table 2. *Interatomic distances* (Å)

All C–H bonds are 1·080 Å.

S(1)–N(23)	1·653 (6)	N(23)–H(24)	0·94 (9)
N(23)–C(5)	1·394 (9)	N(25)–H(26)	1·16 (9)
C(5)–N(25)	1·323 (8)	C(28)–C(30)	1·377 (11)
N(25)–C(6)	1·468 (9)	C(30)–C(32)	1·428 (11)
C(6)–C(9)	1·521 (11)	C(32)–C(33)	1·387 (11)
C(9)–C(12)	1·544 (12)	C(33)–C(35)	1·383 (11)
C(12)–C(15)	1·452 (19)	C(35)–C(27)	1·397 (10)
S(1)–O(2)	1·441 (5)	C(27)–C(28)	1·384 (10)
S(1)–O(3)	1·432 (5)	C(27)–S(1)	1·760 (7)
C(5)–O(4)	1·262 (8)	C(32)–C(19)	1·522 (11)

Table 3. *Bond angles* (°)

C(27) C(28)–C(30)	120·1 (7)	N(25) C(6)–C(9)	112·7 (6)
C(28) C(30) C(32)	119·9 (8)	C(6) C(9)–C(12)	114·5 (8)
C(30) C(32)–C(33)	118·3 (8)	C(9) C(12)–C(15)	106·7 (1·2)
C(32) C(33)–C(35)	122·0 (8)	H(7) C(6)–H(8)	109·5 (0)
C(33) C(35) C(27)	118·4 (8)	H(10) C(9)–H(11)	109·5 (0)
C(35) C(27) C(28)	121·2 (7)	H(13) C(12)–H(14)	109·5 (0)
C(27) C(28)–H(29)	119·3 (4)	H(16) C(15) H(17)	109·5 (0)
C(30)–C(28) H(29)	120·6 (5)	H(17)- C(15)–H(18)	109·5 (0)
C(28) C(30)–H(31)	120·1 (5)	H(18)–C(15)–H(16)	109·5 (0)
C(32)–C(30)–H(31)	120·0 (5)	N(25)–C(6)–C(7)	108·6 (4)
C(32)–C(33)–H(34)	118·6 (5)	N(25) -C(6) H(8)	109·1 (4)
C(35)–C(33) H(34)	119·4 (5)	C(9) C(6)–H(7)	109·3 (6)
C(33)–C(35) H(36)	121·3 (5)	C(9)–C(6)–H(8)	107·7 (5)
C(27) C(35)–H(36)	120·2 (5)	C(6)–C(9)–C(10)	108·9 (6)
C(30)–C(32)–C(19)	120·8 (9)	C(6)–C(9)–H(11)	107·4 (5)
C(33) C(32)–C(19)	120·8 (8)	C(12)–C(9)- H(10)	109·2 (6)
C(32)–C(19)–H(20)	116·7 (6)	C(12)–C(9)–H(11)	107·3 (7)
C(32) C(19)–H(21)	99·4 (5)	C(9) C(12) H(13)	111·0 (6)
C(32) C(19) H(22)	111·8 (5)	C(9) C(12)–H(14)	111·3 (6)
H(20) C(19) H(21)	109·5 (0)	C(15) C(12)–H(13)	111·0 (1·0)
H(21) C(19) H(22)	109·5 (0)	C(15) C(12)–H(14)	107·2 (8)
H(22) C(19) H(20)	109·5 (0)	C(12) C(15) H(16)	101·3 (1·0)
C(28) C(27) S(1)	120·0 (5)	C(12) -C(15) H(17)	128·8 (1·0)
C(35) C(27) S(1)	118·7 (6)	C(12) C(15) H(18)	96·9 (8)
C(27) S(1) N(23)	104·6 (3)	C(5)–N(23)–S(1)	121·1 (5)
C(27) S(1)–O(2)	108·6 (3)	N(23) C(5) -O(4)	120·2 (6)
C(27) S(1) O(3)	109·5 (3)	O(4) C(5)–N(25)	124·1 (7)
O(2) S(1) O(3)	119·0 (3)	N(25)- C(5)–N(23)	115·7 (6)
O(2) S(1) N(23)	110·8 (3)	C(5)–N(25)–H(26)	115 (4)
O(3) S(1)–N(23)	103·4 (3)	H(26) N(25) C(6)	123 (4)
H(24) N(23)- C(5)	117 (5)	C(6) N(25) C(5)	121·6 (6)
S(1) -N(23) H(24)	117 (5)		

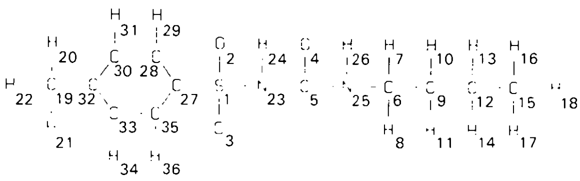

Fig. 1. Diagram of molecule showing numbering of atoms.

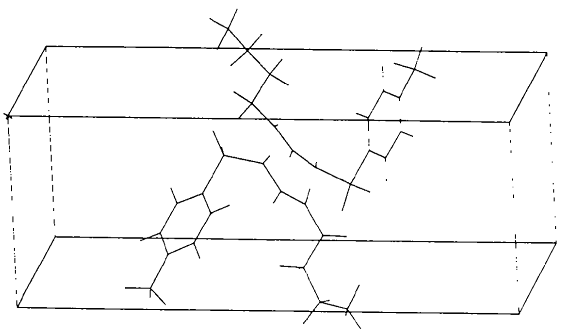

Fig. 2. Stick diagram of two molecules in the unit cell.

Tables 1, 2, and 3 give the atomic coordinates, bond distances and bond angles. Numbers in parentheses represent the e.s.d.'s in units of the last digit. Fig. 1 shows the numbering of the atoms.*

Discussion. The four molecules in the unit cell occur in pairs. Fig. 2 shows two of the molecules in the unit cell, and it can be seen that adjacent cells will have corresponding molecules in close proximity to those in this cell, thus forming a chain through the crystal. The remaining two molecules form a similar chain. Hydrogen bonding may occur between the molecules in three places. Distances between the atoms concerned are $O(2^{i})\cdots N(25^{ii})$ 3·063, $O(4^{i})\cdots N(25^{ii})$ 2·958, $O(4^{i})\cdots N(23^{ii})$ 2·714 Å. According to commonly accepted views on hydrogen bonding, these distances show that such bonding is quite likely. As it has been shown to be relatively easy to convert form *A* to the monoclinic form *B*, *e.g.* by heating (Leary, Ross & Thomas, 1981), it is suggested that these chains are maintained with some reorientation in the transition.

Note added in proof: While this paper was in the press, an analysis of the structure of tolbutamide appeared in this journal (Nirmala & Sake Gowda, 1981) (N & SG). Regrettably, we are forced to disagree with many of their conclusions, and to attribute most of the disagreement to the lower quality of their data.

Our structure is based on 988 reflections collected on a diffractometer, and the residual is 0·048; theirs is based on 615 reflections whose intensities were estimated visually, leading to a residual of 0·083.

Table 4 lists those bond lengths and bond angles for which our values differ from theirs by more than their stated e.s.d.'s, and Table 5 lists the differences between our and their positions of the non-hydrogen atoms. Their work gives no results for the hydrogen atoms.

Table 4. *Comparison of bond lengths* (Å) *and bond angles* (°) *obtained in the two analyses*

This work		N & SG, corresponding result	
S(1)–N(23)	1·653 (6)	S–N(2)	1·73 (2)
N(23)–C(5)	1·394 (9)	N(2)–C(5)	1·52 (4)
C(5)–N(25)	1·323 (8)	C(5)–N(1)	1·38 (4)
N(25)–C(6)	1·468 (9)	N(1)–C(4)	1·51 (3)
C(6)–C(9)	1·521 (11)	C(4)–C(3)	1·62 (4)
C(9)–C(12)	1·544 (12)	C(3)–C(2)	1·68 (5)
C(12)–C(15)	1·452 (19)	C(2)–C(1)	1·28 (8)
C(5)–O(4)	1·262 (8)	C(5)–O(3)	1·11 (5)
C(27)–S(1)	1·760 (7)	C(6)–S	1·68 (2)
C(30)–C(32)–C(19)	120·8 (9)	C(8)–C(9)–C(12)	109 (2)
C(33)–C(32)–C(19)	120·8 (8)	C(10)–C(9)–C(12)	130 (2)
O(2)–S(1)–N(23)	110·8 (3)	O(2)–S–N(2)	116 (1)
O(3)–S(1)–N(23)	103·4 (3)	O(1)–S–N(2)	96 (1)
N(25)–C(6)–C(9)	112·7 (6)	N(1)–C(4)–C(3)	105 (2)
C(9)–C(12)–C(15)	106·7 (1·2)	C(1)–C(2)–C(3)	95 (4)
C(3)–N(23)–S(1)	121·1 (5)	C(5)–N(2)–S	110 (2)
N(23)–C(5)–O(4)	120·2 (6)	N(2)–C(5)–O(3)	125 (3)
O(4)–C(5)–N(25)	124·1 (7)	O(3)–C(5)–N(1)	132 (3)
N(25)–C(5)–N(23)	115·7 (6)	N(2)–C(5)–N(1)	102 (3)
C(6)–N(25)–C(5)	121·6 (6)	C(5)–N(1)–C(4)	110 (2)

Table 5. *Discrepancies in non-hydrogen fractional atom positions obtained in the two analyses*

The discrepancies between the fractional atomic positions are given in a form such that, where the coordinate found in this work is larger, the sign of the discrepancy is positive. Atom and coordinate designations in square brackets are those of Nirmala & Sake Gowda.

Atom designation				
This work	N & SG	x[x]	y[z]	z[y]
S(1)	S[1]	+0·5012	+0·0057	−0·3413
O(2)	O[2]	+0·5010	+0·0012	−0·3443
O(3)	O[1]	+0·5008	+0·0152	−0·3481
O(4)	O[3]	+0·5017	+0·0044	−0·3518
C(5)	C[5]	+0·4998	+0·0063	−0·3356
C(6)	C[4]	+0·4957	−0·0133	−0·3352
C(9)	C[3]	+0·4985	−0·0016	−0·3420
C(12)	C[2]	+0·4980	+0·0166	−0·3353
C(15)	C[1]	+0·5011	+0·0287	−0·3312
C(19)	C[12]	+0·5022	+0·0043	−0·3941
C(27)	C[6]	+0·4986	−0·0016	−0·3454
C(28)	C[7]	+0·5009	−0·0043	−0·3508
C(30)	C[8]	+0·5033	−0·0048	−0·3537
C(32)	C[9]	+0·5026	−0·0044	−0·3563
C(33)	C[10]	+0·5002	+0·0027	−0·3464
C(35)	C[11]	+0·4989	+0·0038	−0·3446
N(23)	N[2]	+0·5009	+0·0092	−0·3614
N(25)	N[1]	+0·5007	+0·0013	−0·3528

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36116 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4 shows that several of the bond lengths obtained by Nirmala & Sake Gowda are most unlikely to be correct, conflicting as they do with generally accepted values for such bonds. Indeed, they draw attention to the unexpected value which they obtained for their C—S distance. Among other anomalies are their C—O distance in the urea group, of 1·11 Å, which is actually less than the bond length in carbon monoxide, and the C—C bond lengths in the *n*-butyl chain, which are not at all what one would expect (1·62, 1·68, 1·28 Å).

The bond angles show similar discrepancies, and it is noteworthy that they find large differences between the sizes of pairs of angles which one would expect to be nearly equal. The clearest example of this is at the phenyl carbon attached to the methyl group, where their angles are given as 109 and 130° whereas ours are both 120·8°. Again, there are some very odd angles in the chain, particularly their C(1)—C(2)—C(3) which is 95°. So large a discrepancy compared with the expected near-tetrahedral value is surely worthy of comment. Our value of 106·7° is more in accord with expectation. Finally, the considerable discrepancies in the fractional atomic positions can be rationalized to some extent by transforming any point (x,y,z) in our structure to a corresponding point $[x-\frac{1}{2}, z+(\sim\frac{1}{3}), y]$ in theirs, but there are still many disagreements which are well outside the e.s.d.'s, particularly with regard to the carbon atom of the *p*-methyl group. The conclusion reached by Nirmala & Sake Gowda about the orientation of this carbon atom with respect to the ring is not sustainable in the light of these inaccuracies.

It is worth pointing out that the solvent which they used for recrystallization is not one mentioned in the standard papers on preparation, and no analysis figures were given. In view of this, it cannot be regarded as certain that the two analyses of the structure actually refer to the same material.

References

LEARY, J. R., ROSS, S. D. & THOMAS, M. K. J. (1981). *Pharm. Weekbl.* (*Sci. Ed.*), **3**, 62–66.

MACEK, T. J. (1954). US patent 2671750.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J. P. & WOOLFSON, M. M. (1978). *MULTAN 78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MAKHNENKO, N. I. & SYSOEVA, T. F. (1959). *Zh. Prikl. Khim.* (*Moscow*), **32**, 449–450.

MULLER, B. W. & LAGAS, M. (1979). *Pharm. Weekbl.* (*Sci. Ed.*), **1**, 449–456.

NIRMALA, K. A. & SAKE GOWDA, D. S. (1981). *Acta Cryst.* B**37**, 1597–1599.

SHELDRICK, G. M. (1976). *SHELX*. A program for crystal structure determination. Univ. of Cambridge, England.

SIMMONS, D. L., RANZ, R. J., GYANCHANDANI, N. D. & PICOTTE, P. (1972). *Can. J. Pharm. Sci.* **7**, 121–123.

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4-Chloro-3-{[chloro(1-pyrrolidinyl)methylene]amino}-3-cyclobutene-1,2-dione

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Abstract. $C_9H_8Cl_2N_2O_2$, orthorhombic, $P2_1ca$, a = 7·108 (2), b = 9·602 (4), c = 15·493 (6) Å, V = 1057·3 (6) Å^3, Z = 4, D_c = 1·552 Mg m^{-3}, 993 independent reflections with $\sin\theta/\lambda \leq 0{\cdot}60$ Å^{-1}, $R(F)$ = 0·039, $R_w(F)$ = 0·030. The molecule has the Z configuration. The angle between the nearly planar cyclobutenedione group and the amidine group is 50 (1)°.

Introduction. The synthesis of the title compound was described by Ried, Dietschmann & Erle (1981).

A transparent, yellow, plate-shaped crystal with dimensions 0·15 × 0·25 × 0·60 mm was selected for the experiments. Precession photographs showed the space group to be either $P2_1ca$ or $Pmca$. The former was confirmed during the structure determination.* Data were collected on a Syntex $P2_1$ diffractometer with Mo $K\alpha$ radiation. Reflections in three octants were

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* $P2_1ca$ corresponds to a non-standard setting of space group $Pca2_1$. The coordinates of the equivalent positions are: x,y,z; $\frac{1}{2}+x, -y, -z$; $\frac{1}{2}+x, y, \frac{1}{2}-z$; $x, -y, \frac{1}{2}+z$.

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