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.

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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, $P2_1ca$, $a = 7{\cdot}108\,(2)$, $b = 9{\cdot}602\,(4)$, $c = 15{\cdot}493\,(6)$ Å, $V = 1057{\cdot}3\,(6)$ Å^3, $Z = 4$, $D_c = 1{\cdot}552$ Mg m^{-3}, 993 independent reflections with $\sin\theta/\lambda \le 0{\cdot}60$ Å^{-1}, $R(F) = 0{\cdot}039$, $R_w(F) = 0{\cdot}030$. 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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measured up to $\sin\theta/\lambda = 0{\cdot}60$ Å^{-1}, resulting in 3021 reflections, of which 1029 were independent. Background corrections were made as described by Blessing, Coppens & Becker (1974). Three standard reflections, remeasured after every 65 reflections, showed a gradual decrease of about 8% during the measurements. This effect was corrected by scaling the reflections with respect to the standards. An absorption correction $[\mu(\text{Mo } K\alpha) = 0{\cdot}595 \text{ mm}^{-1}]$ gave transmission factors ranging from 0·87 to 0·92. Weights were assigned according to $w(I) = [\sigma^2(I)_{\text{counting}} + (0{\cdot}03I)^2]^{-1}$. The equivalent reflections were averaged. 993 resulting reflections had $I > 0$ and were used in the structure determination and refinement. The structure was determined with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974). The H atoms were located from a difference synthesis. The structure refined to $R(F) = 0{\cdot}039$ and $R_w(F) = 0{\cdot}030$. Scattering factors were from *International Tables for X-ray Crystallography* (1974), except those for H which were from Stewart, Davidson & Simpson (1965). The calculations were carried out with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) on the Univac 1108 computer of the University of Frankfurt. The atomic coordinates are reported in Table 1.*

Table 1. *Positional parameters and equivalent isotropic values of the anisotropic thermal parameters* ($U_{\text{eq}} = \frac{1}{3}$ *trace* $\tilde{\mathbf{U}}$) *for non-*H *atoms and isotropic thermal parameters for* H

	x	y	z	U_{eq} (Å^2)
Cl(1)	0·0	0·35128 (10)	0·14391 (5)	0·052 (1)
Cl(2)	−0·1260 (2)	0·66140 (9)	0·42972 (6)	0·050 (1)
C(1)	−0·0092 (6)	0·3431 (3)	0·2525 (2)	0·037 (2)
C(2)	−0·0676 (5)	0·2332 (4)	0·3124 (2)	0·046 (2)
C(3)	−0·0161 (6)	0·3360 (3)	0·3856 (2)	0·046 (2)
C(4)	0·0347 (5)	0·4401 (3)	0·3155 (2)	0·037 (2)
C(5)	0·0793 (5)	0·6630 (4)	0·3681 (2)	0·037 (2)
C(6)	0·3678 (7)	0·7836 (4)	0·3304 (3)	0·050 (2)
C(7)	0·4601 (6)	0·9104 (4)	0·3731 (3)	0·057 (3)
C(8)	0·2956 (7)	0·9986 (5)	0·3999 (3)	0·056 (3)
C(9)	0·1493 (7)	0·8961 (4)	0·4311 (3)	0·046 (2)
O(1)	−0·1291 (5)	0·1164 (3)	0·3084 (2)	0·067 (2)
O(2)	−0·0105 (5)	0·3343 (3)	0·4623 (2)	0·068 (2)
N(1)	0·1121 (4)	0·5662 (3)	0·3107 (2)	0·038 (2)
N(2)	0·1862 (5)	0·7719 (3)	0·3774 (2)	0·038 (2)
H(1)	0·337 (6)	0·804 (3)	0·267 (2)	0·054 (11)
H(2)	0·448 (7)	0·698 (5)	0·344 (3)	0·088 (16)
H(3)	0·540 (7)	0·877 (4)	0·431 (3)	0·063 (12)
H(4)	0·556 (8)	0·948 (5)	0·328 (3)	0·109 (16)
H(5)	0·313 (6)	1·069 (4)	0·442 (2)	0·066 (12)
H(6)	0·232 (7)	1·040 (5)	0·351 (3)	0·091 (16)
H(7)	0·165 (6)	0·866 (3)	0·492 (3)	0·056 (11)
H(8)	0·026 (6)	0·925 (4)	0·419 (2)	0·046 (10)

Discussion. A stereoscopic view of the molecule is shown in Fig. 1; bond lengths and angles are given in Table 2. The central part of the molecule has the *Z* configuration. The bond lengths can be explained on the basis of the three resonance structures shown in Fig. 2. The electron delocalization over a large part of the molecule would lead to a considerable thermal stability of the compound. This has been experimentally confirmed (Ried *et al.*, 1981).

The molecule contains three almost planar parts (Table 3). The cyclobutene group makes an angle of

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

C(1)–C(2)	1·466 (5)	N(1)–C(5)	1·307 (4)
C(1)–C(4)	1·384 (5)	C(5)–N(2)	1·301 (5)
C(1)–Cl(1)	1·686 (3)	C(5)–Cl(2)	1·743 (4)
C(2)–C(3)	1·547 (5)	N(2)–C(6)	1·486 (6)
C(2)–O(1)	1·205 (5)	N(2)–C(9)	1·477 (5)
C(3)–C(4)	1·519 (5)	C(6)–C(7)	1·532 (6)
C(3)–O(2)	1·189 (4)	C(7)–C(8)	1·502 (7)
C(4)–N(1)	1·332 (4)	C(8)–C(9)	1·512 (7)
		$\{C{-}H\}_{\text{average}}$	1·01 (2)
C(2)–C(1)–C(4)	95·8 (3)	C(4)–N(1)–C(5)	122·3 (3)
C(2)–C(1)–Cl(1)	132·6 (3)	N(1)–C(5)–N(2)	122·8 (3)
C(4)–C(1)–Cl(1)	131·6 (3)	N(1)–C(5)–Cl(2)	121·0 (3)
C(1)–C(2)–C(3)	86·4 (3)	N(2)–C(5)–Cl(2)	115·8 (3)
C(1)–C(2)–O(1)	137·7 (3)	C(5)–N(2)–C(6)	121·0 (3)
C(3)–C(2)–O(1)	135·9 (3)	C(5)–N(2)–C(9)	127·4 (3)
C(2)–C(3)–C(4)	87·3 (3)	C(6)–N(2)–C(9)	111·7 (3)
C(2)–C(3)–O(2)	137·0 (3)	N(2)–C(6)–C(7)	102·8 (3)
C(4)–C(3)–O(2)	135·7 (3)	C(6)–C(7)–C(8)	103·6 (4)
C(1)–C(4)–C(3)	90·5 (3)	C(7)–C(8)–C(9)	104·9 (4)
C(1)–C(4)–N(1)	131·7 (3)	C(8)–C(9)–N(2)	102·9 (4)
C(3)–C(4)–N(1)	137·4 (3)		

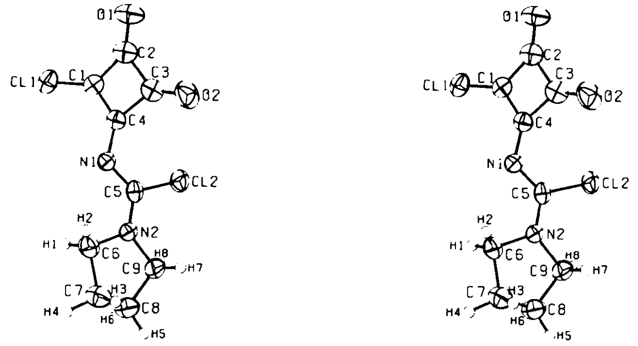

Fig. 1. Stereoscopic view of the molecule and numbering scheme. The thermal ellipsoids correspond to the 50% probability surfaces; H atoms are drawn on an arbitrary scale.

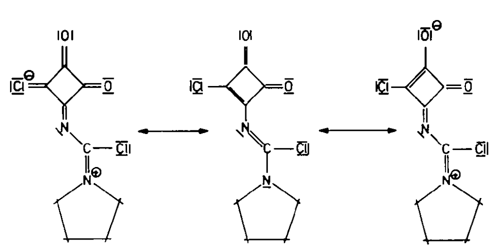

Fig. 2. The main resonance structures of the title compound.

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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 36173 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Distances* (Å) *from least-squares planes*

I		II		III	
C(1)	0·009 (4)	C(5)	0·034 (4)	C(5)	−0·002 (4)
C(2)	−0·008 (4)	Cl(2)	−0·009 (1)	N(2)	0·005 (9)
C(3)	0·008 (4)	N(1)	−0·013 (3)	C(6)	−0·001 (6)
C(4)	−0·009 (4)	N(2)	−0·012 (3)	C(9)	−0·001 (6)
Cl(1)*	0·025 (1)	C(4)*	0·375 (4)		
O(1)*	−0·033 (4)				
O(2)*	0·063 (4)				
N(1)*	0·089 (3)				

Angles between planes (°): I–II 50 (1)
II–III 5 (1)

* Atoms not used in the definition of the least-squares plane.

50 (1)° with the amidine group. This rotation is necessary to avoid repulsion between Cl(2) and O(2). The observed distance is 3·286 (6) Å and corresponds to the sum of the van der Waals radii of Cl and O. The angle between the cyclobutene and amidine groups results from torsion angles C(3)—C(4)—N(1)—C(5) of −37 (1)° and C(4)—N(1)—C(5)—Cl(2) of −24 (1)°. This conformation allows an almost equal contribution of the N(1) π electron to the neighbouring π systems. C(5) has a small amount of pyramidal character (Table 3).

In the crystal structure there is an intermolecular contact distance of 3·440 (4) Å between Cl(1) and Cl(2) (symmetry code: x, $1 - y$, $-\frac{1}{2} + z$). No other intermolecular distances are shorter than the sum of the van der Waals radii of the constituent atoms.

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Notes and News

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ICSD—Inorganic Crystal Structure Data Base

Complementary to the well known Cambridge Crystallographic Data File, a similar file for inorganic substances has been established at the University of Bonn (G. Bergerhoff and I. D. Brown). The current file contains information on 9000 structures: chemical name, chemical formula, mineral name, unit cell, space group, coordinates, temperature factors, references, remarks. More detailed information will be given in a paper to be published in *Acta Crystallographica*. The data base will be made available by Fachinformationszentrum Energie Physik Mathematik GmbH (Dr H. Behrens), D-7514 Eggenstein-Leopoldshafen, Federal Republic of Germany, from 1982 on, in three versions:

(1) On-line access *via* telecommunications systems (Euronet, Datex-P, *etc.*).

(2) Leasing the up-to-date data base and retrieval programs (IBM–FORTRAN) at an annual rate.

(3) Leasing only the up-to-date data base at an annual rate.

Detailed conditions are available on request from Dr Behrens at the address given above.