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The Crystal Structure of 2-Methyl-3-phenyl-4-(*N*-methyl-*N*-hydroxyamidin)-isoxazolin-5-one Hydrobromide

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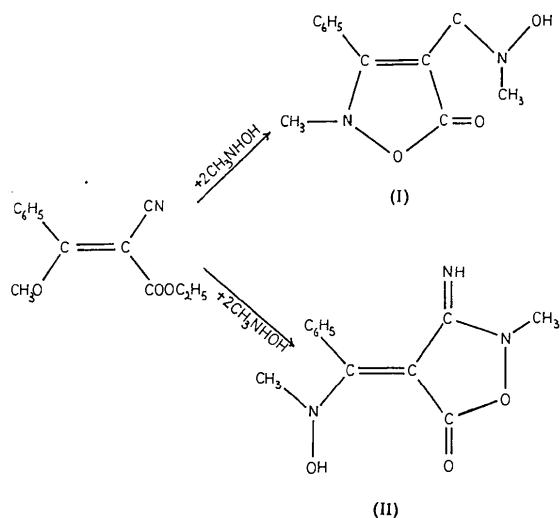
Crystals of 2-methyl-3-phenyl-4-(*N*-methyl-*N*-hydroxyamidin)isoxazolin-5-one hydrobromide are orthorhombic, space group *Pbca*, with $a = 19.539$ (2), $b = 10.676$ (2) and $c = 13.418$ (2) Å. There are eight formula units per unit cell. The structure was solved by the heavy atom method employing 2954 intensity data collected on a Weissenberg apparatus. It was refined by least-squares methods with anisotropic thermal parameters for all the non-hydrogen atoms to an *R* index of 0.075 for 1452 independent observed reflexions. Bond distances are in reasonable agreement with the corresponding values for related substances. The nitrogen atom of the iminic group is protonated. The planarity of the *N*-methyl-*N*-hydroxyamidin chain indicates the occurrence of two mesomeric forms for this group.

Introduction

The present X-ray study was undertaken in order to determine the molecular structure of the product of

the reaction between ethyl- α -cyano- β -methoxy- β -phenyl acrylate and an excess of methylhydroxylamine because spectroscopic data and chemical behaviour did not confirm which of the two stereoisomers (**I**) and

(II), among several possible compounds, was formed (Adembri & Tedeschi, 1967). The two chemical reactions can be sketched as it follows:



Experimental

Crystals of the hydrobromide of the reaction compound were kindly supplied by Professor Tedeschi of the Institute of Organic Chemistry of Florence University. They are short, prismatic in habit and colourless, but turn brown after a long exposure to the air and to X-rays. Their melting point is 204–206°C.

From preliminary oscillation and Weissenberg photographs, crystals were assigned to the orthorhombic system, space group *Pbca*, and approximate lattice parameters were determined. Cell constants were refined by a least-squares method employing 2θ values of 37 high Bragg-angle reflexions, measured on *hk0* and *h0l* Weissenberg photographs.

The density, measured by flotation in an aqueous solution of potassium iodomercurate, is 1.53 g.cm⁻³, in agreement with the calculated value of 1.556 g.cm⁻³ if eight molecules are assumed present in the unit cell. Crystal data are reported in Table 1.

Table 1. *Crystal data for 2-methyl-3-phenyl-4-(N-methyl-N-hydroxyamidin)isoxazolin-5-one hydrobromide*

F.W.: 328·1

Melting point: 205 ± 1°C

Systematic absences: *0kl* *k* = 2*n* + 1; *h0l* *l* = 2*n* + 1;
hk0 *h* = 2*n* + 1

Space group: *Pbca*

a = 19.539 ± 0.002 Å
b = 10.676 ± 0.002

c = 13.418 ± 0.002

D_m = 1.53 g.cm⁻³

F(000) = 1328

V = 2799 Å³

Z = 8

D_x = 1.556 g.cm⁻³

μ(Cu *K*α) = 45.0 cm⁻¹.

For the structural study, reflexions from the *hk0* to *hk10* layers were collected from a well formed crys-

tal elongated in the *c* direction with dimensions 0.8 × 0.4 × 0.3 mm. Since this crystal turned brown during X-ray exposure, intensity data from the *h0l* to *h7l* layers were collected from a second crystal with a different shape and approximate dimensions 0.6 × 0.2 × 0.3 mm.

An integrating Weissenberg apparatus was used employing equiinclination and multiple-film techniques and Cu *K*α radiation. 2954 independent diffraction effects were obtained, of which 1502 were too weak to be measured. These reflexions were given an intensity value of zero.

Intensities were measured with a microdensitometer and converted to *F*² values, corrected by the Lorentz and polarization factors. An approximate cylindrical absorption correction was applied to the *hk0* ~ *hk10* data set. A correction according to the method of De Meuleenaer & Tompa (1965) was applied to *h0l* ~ *h7l* data.

In order to obtain a unique set of data, the scaling of the *F*²'s from different layers was carried out by the correlation of the common reflexions, employing the least-squares method of Rae (1965).

Determination and refinement

The structure was solved by the heavy atom method; the coordinates of the bromine atom were determined from a three-dimensional Patterson function and a complete, but approximate, structural model, with the exclusion of the hydrogen atoms, was derived by means of successive electron density maps leading to a reliability index of 0.27. This model was consistent with the chemical formula (I).

The structure was refined by the least-squares method. The weighting scheme used in the refinement was $\frac{1}{w} = 1$ for observed reflexions with $|F_o| \leq 4|F_{min}|$, $\frac{1}{w} = 4|F_{min}|/|F_o|$ for observed reflexions with $|F_o| > 4|F_{min}|$. After five cycles of calculation, using the block-diagonal approximation and individual isotropic thermal parameters, the *R* index dropped to 0.14. At this stage two more cycles, with anisotropic thermal parameters, were computed. The *R* index decreased to 0.12.

A difference Fourier synthesis was then computed, which revealed residual peaks at the positions expected for the hydrogen atoms. In the last cycle, the coordinates of all the atoms and the anisotropic thermal parameters of the Br, C, O, N atoms were refined (an isotropic value of 5 Å² was set for all the hydrogen atoms). The final *R* value was 0.075 for all the observed reflexions.

The atomic scattering factors used in the calculations were taken from *International Tables for X-ray Crystallography* (1962).

Observed and calculated structure factors are given in Table 2. Final atomic coordinates and thermal parameters with estimated standard deviations are listed in Tables 3 and 4 respectively.

Table 2. Observed and calculated structure factors ($\times 10$)

Unobserved reflexions were given zero intensities. The asterisks indicate reflexions affected by extinction.

Table 2 (cont.)

4	0	18	6	555	555	7	322	320	11	272	250	11	0	0	0	0
5	0	19	2	141	151	8	741	741	11	251	262	11	120	149	0	0
7	423	411	7	175	173	10	671	203	0	213	245	10	234	258	11	289
8	226	221	8	201	201	11	345	445	2	125	345	10	216	151	0	213
10	140	275	15	0	0	16	77	137	32	3	26	15	100	236	15	21
11	175	385	15	157	157	16	311	310	2	249	229	11	137	153	15	221
12	122	122	12	122	105	16	151	175	5	21	16	15	189	189	15	237
13	272	1025	15	158	116	16	151	175	6	138	16	15	189	189	15	237
14	160	160	15	151	121	12	150	151	7	175	175	16	15	111	0	12
15	138	350	16	151	121	12	150	151	8	175	175	16	15	111	0	12
16	185	185	16	185	121	12	180	181	10	167	167	9	128	185	16	185
17	222	222	20	20	07	21	210	210	11	205	205	10	160	205	20	210
18	167	167	2	167	92	21	165	165	13	193	193	2	140	207	16	207
19	123	166	4	166	166	6	165	165	14	165	165	5	140	207	16	207
20	149	190	6	190	166	7	185	185	15	185	185	6	140	207	16	207
21	218	211	9	210	185	10	185	185	16	185	185	7	140	207	16	207
22	137	187	10	187	166	11	185	185	17	185	185	8	140	207	16	207
23	125	187	10	187	166	11	185	185	18	185	185	9	140	207	16	207
24	172	187	10	187	166	11	185	185	19	185	185	10	140	207	16	207
25	108	187	10	187	166	11	185	185	20	185	185	11	140	207	16	207
26	125	187	10	187	166	11	185	185	21	185	185	12	140	207	16	207
27	147	187	10	187	166	11	185	185	22	185	185	13	140	207	16	207
28	167	187	10	187	166	11	185	185	23	185	185	14	140	207	16	207
29	125	187	10	187	166	11	185	185	24	185	185	15	140	207	16	207
30	172	187	10	187	166	11	185	185	25	185	185	16	140	207	16	207
31	125	187	10	187	166	11	185	185	26	185	185	17	140	207	16	207
32	172	187	10	187	166	11	185	185	27	185	185	18	140	207	16	207
33	125	187	10	187	166	11	185	185	28	185	185	19	140	207	16	207
34	172	187	10	187	166	11	185	185	29	185	185	20	140	207	16	207
35	125	187	10	187	166	11	185	185	30	185	185	21	140	207	16	207
36	172	187	10	187	166	11	185	185	31	185	185	22	140	207	16	207
37	125	187	10	187	166	11	185	185	32	185	185	23	140	207	16	207
38	172	187	10	187	166	11	185	185	33	185	185	24	140	207	16	207
39	125	187	10	187	166	11	185	185	34	185	185	25	140	207	16	207
40	172	187	10	187	166	11	185	185	35	185	185	26	140	207	16	207
41	125	187	10	187	166	11	185	185	36	185	185	27	140	207	16	207
42	172	187	10	187	166	11	185	185	37	185	185	28	140	207	16	207
43	125	187	10	187	166	11	185	185	38	185	185	29	140	207	16	207
44	172	187	10	187	166	11	185	185	39	185	185	30	140	207	16	207
45	125	187	10	187	166	11	185	185	40	185	185	31	140	207	16	207
46	172	187	10	187	166	11	185	185	41	185	185	32	140	207	16	207
47	125	187	10	187	166	11	185	185	42	185	185	33	140	207	16	207
48	172	187	10	187	166	11	185	185	43	185	185	34	140	207	16	207
49	125	187	10	187	166	11	185	185	44	185	185	35	140	207	16	207
50	172	187	10	187	166	11	185	185	45	185	185	36	140	207	16	207
51	125	187	10	187	166	11	185	185	46	185	185	37	140	207	16	207
52	172	187	10	187	166	11	185	185	47	185	185	38	140	207	16	207
53	125	187	10	187	166	11	185	185	48	185	185	39	140	207	16	207
54	172	187	10	187	166	11	185	185	49	185	185	40	140	207	16	207
55	125	187	10	187	166	11	185	185	50	185	185	41	140	207	16	207
56	172	187	10	187	166	11	185	185	51	185	185	42	140	207	16	207
57	125	187	10	187	166	11	185	185	52	185	185	43	140	207	16	207
58	172	187	10	187	166	11	185	185	53	185	185	44	140	207	16	207
59	125	187	10	187	166	11	185	185	54	185	185	45	140	207	16	207
60	172	187	10	187	166	11	185	185	55	185	185	46	140	207	16	207
61	125	187	10	187	166	11	185	185	56	185	185	47	140	207	16	207
62	172	187	10	187	166	11	185	185	57	185	185	48	140	207	16	207
63	125	187	10	187	166	11	185	185	58	185	185	49	140	207	16	207
64	172	187	10	187	166	11	185	185	59	185	185	50	140	207	16	207
65	125	187	10	187	166	11	185	185	60	185	185	51	140	207	16	207
66	172	187	10	187	166	11	185	185	61	185	185	52	140	207	16	207
67	125	187	10	187	166	11	185	185	62	185	185	53	140	207	16	207
68	172	187	10	187	166	11	185	185	63	185	185	54	140	207	16	207
69	125	187	10	187	166	11	185	185	64	185	185	55	140	207	16	207
70	172	187	10	187	166	11	185	185	65	185	185	56	140	207	16	207
71	125	187	10	187	166	11	185	185	66	185	185	57	140	207	16	207
72	172	187	10	187	166	11	185	185	67	185	185	58	140	207	16	207
73	125	187	10	187	166	11	185	185	68	185	185	59	140	207	16	207
74	172	187	10	187	166	11	185	185	69	185	185	60	140	207	16	207
75	125	187	10	187	166	11	185	185	70	185	185	61	140	207	16	207
76	172	187	10	187	166	11	185	185	71	185	185	62	140	207	16	207
77	125	187	10	187	166	11	185	185	72	185	185	63	140	207	16	207
78	172	187	10	187	166	11	185	185	73	185	185	64	140	207	16	207
79	125	187	10	187	166	11	185	185	74	185	185	65	140	207	16	207
80	172	187	10	187	166	11	185	185	75	185	185	66	140	207	16	207
81	125	187	10	187	166	11	185	185	76	185	185	67	140	207	16	207
82	172	187	10	187	166	11	185	185	77	185	185	68	140	207	16	207
83	125	187	10	187	166	11	185	185	78	185	185	69	140	207	16	207
84	172	187	10	187	166	11	185	185	79	185	185	70	140	207	16	207
85	125	187	10	187	166	11	185	185	80	185	185	71	140	207	16	207
86	172	187	10	187	166	11	185	185	81	185	185	72	140	207	16	207
87	125	187	10	187	166	11	185	185	82	185	185	73	140	207	16	207
88	172	187	10	187	166	11	185	185	83	185	185	74	140	207	16	207
89	125	187	10	187	166	11	185	185	84	185	185	75	140	207	16	207
90	172	187	10	187	166	11	185	185	85	185	185	76	140	207	16	207
91	125	187	10	187	166	11	185	185	86	185	185	77	140	207	16	207
92	172	187	10	187	166	11	185	185	87	185	185	78	140	207	16	207
93	125	187	10	187	166	11	185	185	88	185	185	79	140	207	16	207
94	172	187	10	187	166	11	185	185	89	185	185	80	140	207	16	207
95	125	187	10	187	166	11	185	185	90	185	185	81	140	207	16	207
96	172	187	10	187	166											

Table 3. Fractional atomic coordinates with standard deviations in parentheses

Atomic coordinates are ($\times 10^4$) for non-hydrogen and ($\times 10^3$) for hydrogen atoms.

	<i>x</i>	<i>y</i>	<i>z</i>
Br	1353 (1)	1123 (1)	1923 (1)
C(1)	1515 (5)	1624 (10)	4738 (8)
C(2)	984 (6)	792 (10)	4574 (9)
C(3)	950 (6)	-328 (11)	5052 (10)
C(4)	1471 (7)	-648 (12)	5751 (11)
C(5)	1980 (7)	176 (12)	5926 (10)
C(6)	2028 (6)	1319 (11)	5408 (9)
C(7)	1517 (5)	2820 (10)	4223 (8)
N(1)	2096 (4)	3352 (9)	3918 (7)
O(1)	1949 (3)	4537 (7)	3468 (6)
C(8)	1244 (5)	4736 (10)	3564 (9)
C(9)	983 (5)	3622 (9)	4036 (7)
C(10)	268 (5)	3525 (9)	4355 (7)
N(2)	102 (4)	3423 (8)	5304 (6)
N(3)	-217 (4)	3612 (8)	3672 (7)
C(11)	-133 (7)	3496 (13)	2581 (9)
O(2)	-886 (4)	3610 (7)	4006 (6)
C(12)	2716 (5)	2810 (12)	3503 (10)
O(3)	1004 (4)	5707 (7)	3314 (6)
H[C(2)]	60 (6)	89 (12)	408 (9)
H[C(3)]	64 (6)	-107 (11)	503 (10)
H[C(4)]	135 (6)	-167 (12)	615 (9)
H[C(5)]	230 (6)	-29 (12)	631 (9)
H[C(6)]	243 (7)	197 (12)	549 (9)
H(1)[N(2)]	55 (6)	319 (12)	554 (9)
H(2)[N(2)]	-36 (6)	355 (11)	546 (9)
H(1)[C(11)]	33 (6)	308 (12)	254 (10)
H(2)[C(11)]	-23 (6)	442 (12)	236 (11)
H(3)[C(11)]	-45 (6)	265 (12)	246 (10)
H[O(2)]	-109 (6)	465 (12)	398 (9)
H(1)[C(12)]	274 (6)	249 (13)	289 (9)
H(2)[C(12)]	269 (6)	199 (12)	370 (9)
H(3)[C(12)]	310 (6)	319 (12)	383 (9)

Results and discussion

The projection of the structure along the *b* axis is shown in Fig. 1. Bond distances and angles are reported in Fig. 2 and in Table 5.

The heterocyclic ring is planar in the limits of experimental error. The equation of the best plane (plane I) through the five atoms of the ring, referred to the crystallographic axes, is given in Table 6. Deviations from this plane are reported in the same table.

Table 5. Interatomic distances and bond angles with standard deviations in parentheses

(a) Distances	
C(1)—C(2)	1.38 (1) Å
C(2)—C(3)	1.36 (2)
C(3)—C(4)	1.42 (2)
C(4)—C(5)	1.35 (2)
C(5)—C(6)	1.41 (2)
C(6)—C(1)	1.38 (2)
C(1)—C(7)	1.45 (2)
C(7)—N(1)	1.33 (1)
N(1)—C(12)	1.45 (1)
N(1)—O(1)	1.43 (1)
O(1)—C(8)	1.40 (1)
C(8)—O(3)	1.19 (1)
C(8)—C(9)	1.44 (1)
C(9)—C(7)	1.37 (1)
C(9)—C(10)	1.46 (1)
C(10)—N(2)	1.32 (1)
C(10)—N(3)	1.32 (1)
N(3)—O(2)	1.38 (1)
N(3)—C(11)	1.48 (2)
C(2)—H[C(2)]	1.01 (12)
C(3)—H[C(3)]	1.00 (12)
C(4)—H[C(4)]	1.24 (12)
C(5)—H[C(5)]	0.95 (12)
C(6)—H[C(6)]	1.05 (13)
N(2)—H(1)[N(2)]	0.97 (12)
N(2)—H(2)[N(2)]	0.93 (12)
C(11)—H(1)[C(11)]	1.00 (12)
C(11)—H(2)[C(11)]	1.05 (13)
C(11)—H(3)[C(11)]	1.11 (13)
O(2)—H[O(2)]	1.18 (13)
C(12)—H(1)[C(12)]	0.89 (12)
C(12)—H(2)[C(12)]	0.92 (13)
C(12)—H(3)[C(12)]	0.97 (13)

Table 4. Anisotropic thermal parameters ($\times 10^4$) for non-hydrogen atoms

Temperature factors are in the form

$$T = \exp(-h^2\beta_{11} - k^2\beta_{22} - l^2\beta_{33} - 2hk\beta_{12} - 2hl\beta_{13} - 2kl\beta_{23}).$$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Br	30 (1)	136 (1)	61 (1)	-7 (1)	-2 (1)	-6 (1)
C(1)	22 (3)	89 (11)	55 (6)	12 (4)	1 (3)	-5 (7)
C(2)	28 (3)	93 (12)	70 (8)	4 (5)	5 (4)	2 (7)
C(3)	34 (4)	99 (13)	85 (9)	0 (6)	11 (5)	6 (9)
C(4)	53 (5)	98 (13)	80 (9)	15 (7)	12 (6)	3 (9)
C(5)	41 (4)	114 (15)	73 (9)	21 (6)	-7 (5)	25 (9)
C(6)	28 (3)	104 (14)	76 (8)	19 (5)	-1 (4)	-16 (9)
C(7)	22 (3)	97 (12)	51 (6)	3 (4)	2 (3)	-11 (7)
N(1)	22 (2)	90 (10)	81 (7)	3 (4)	2 (3)	-16 (6)
O(1)	23 (2)	78 (8)	79 (6)	0 (3)	10 (3)	3 (5)
C(8)	30 (3)	80 (12)	59 (7)	6 (5)	9 (4)	-5 (7)
C(9)	21 (2)	77 (10)	49 (6)	0 (4)	0 (3)	-5 (6)
C(10)	27 (3)	63 (10)	52 (6)	6 (4)	0 (3)	-5 (6)
N(2)	25 (3)	101 (10)	54 (5)	4 (4)	5 (3)	18 (6)
N(3)	22 (2)	105 (11)	64 (6)	3 (4)	-4 (3)	2 (7)
C(11)	46 (5)	162 (18)	46 (7)	21 (7)	-6 (5)	-15 (9)
O(2)	22 (2)	113 (9)	88 (6)	0 (3)	-1 (3)	7 (6)
C(12)	28 (3)	112 (15)	103 (10)	8 (6)	12 (5)	-7 (10)
O(3)	32 (2)	88 (8)	77 (6)	8 (4)	12 (3)	19 (5)

Table 5 (cont.)

(b) Angles

C(1)—C(2)—C(3)	121.7 (1.1) $^\circ$
C(2)—C(3)—C(4)	119.2 (1.1)
C(3)—C(4)—C(5)	119.0 (1.2)
C(4)—C(5)—C(6)	121.9 (1.2)
C(5)—C(6)—C(1)	118.4 (1.0)
C(6)—C(1)—C(2)	119.7 (1.0)
C(2)—C(1)—C(7)	119.4 (0.9)
C(6)—C(1)—C(7)	120.9 (0.9)
C(1)—C(7)—N(1)	121.6 (0.9)
C(1)—C(7)—C(9)	129.2 (0.9)
N(1)—C(7)—C(9)	108.9 (0.9)
C(7)—N(1)—O(1)	109.7 (0.8)
C(7)—N(1)—C(12)	131.0 (1.0)
C(12)—N(1)—O(1)	111.0 (0.8)
N(1)—O(1)—C(8)	107.0 (0.7)
O(1)—C(8)—C(9)	105.3 (0.8)
O(1)—C(8)—O(3)	119.7 (1.0)
O(3)—C(8)—C(9)	134.9 (1.0)
C(8)—C(9)—C(7)	109.0 (0.9)
C(8)—C(9)—C(10)	121.6 (0.9)
C(7)—C(9)—C(10)	128.9 (0.9)
C(9)—C(10)—N(2)	121.5 (0.9)
C(9)—C(10)—N(3)	118.4 (0.9)
N(2)—C(10)—N(3)	120.0 (0.9)
C(10)—N(3)—O(2)	116.9 (0.8)
C(10)—N(3)—C(11)	127.0 (0.9)
O(2)—N(3)—C(11)	115.2 (0.8)
C(1)———C(2)——H[C(2)]	127 (7) $^\circ$
H[C(2)]——C(2)——C(3)	112 (7)
C(2)——C(3)——H[C(3)]	135 (8)
H[C(3)]——C(3)——C(4)	106 (7)
C(3)——C(4)——H[C(4)]	111 (6)
H[C(4)]——C(4)——C(5)	129 (6)
C(4)——C(5)——H[C(5)]	104 (8)
H[C(5)]——C(5)——C(6)	133 (8)
C(5)——C(6)——H[C(6)]	124 (7)
C(10)——N(2)——H(1) [N(2)]	97 (7)
C(10)——N(2)——H(2) [N(2)]	116 (8)
N(3)——O(2)——H[O(2)]	108 (6)
N(3)——C(11)——H(1) [C(11)]	101 (8)
N(3)——C(11)——H(2) [C(11)]	100 (8)
N(3)——C(11)——H(3) [C(11)]	98 (7)
N(1)——C(12)——H(1) [C(12)]	124 (7)
N(1)——C(12)——H(2) [C(12)]	103 (7)
N(1)——C(12)——H(3) [C(12)]	108 (7)
H(1) [N(2)]——N(2)——H(2) [N(2)]	147 (11)
H(1) [C(11)]——C(11)——H(2) [C(11)]	124 (10)
H(1) [C(11)]——C(11)——H(3) [C(11)]	98 (9)
H(2) [C(11)]——C(11)——H(3) [C(11)]	129 (10)
H(1) [C(12)]——C(12)——H(2) [C(12)]	84 (11)
H(1) [C(12)]——C(12)——H(3) [C(12)]	122 (11)
H(2) [C(12)]——C(12)——H(3) [C(12)]	109 (10)

Bond distances in the isoxazolin-5-one ring are comparable with those found in two other analogous compounds, *i.e.* in *N*-methyl-4-phenylisoxazolin-5-one (Sabelli & Zanazzi, 1969a) and in *N*-methyl-3-phenyl-4-bromoisoaxazolin-5-one (Sabelli & Zanazzi, 1969b).

The phenyl ring is planar; the equation of the best plane (plane II) as well as the atomic deviations from this plane are listed in Table 6. The bonds C(1)—C(7) (1.45 Å) and C(9)—C(10) (1.46 Å) are significantly shorter than a single C—C bond while C(7)—C(9) is longer than a double C=C bond (1.37 Å). This seems to indicate a certain degree of conjugation between

the phenyl ring and the atoms C(7), C(9) and (C10). However, the high value (39°45') of the dihedral angle between plane I and plane II is consistent with only partial conjugation.

Table 6. Least-squares planes

(a) Least-squares plane of the heterocyclic ring

$$2.542x + 4.591y + 11.987z - 6.752 = 0$$

Atoms included in plane	Δ	Atoms not included in plane	Δ
C(7)	-0.01 Å	C(1)	+0.06 Å
N(1)	+0.02	C(10)	+0.15
O(1)	-0.02	O(3)	+0.10
C(8)	+0.01	C(12)	-0.57
C(9)	0.00		

(b) Least-squares plane of the phenyl ring

$$-10.355x + 4.608y + 9.795z - 3.818 = 0$$

C(1)	+0.00 Å	C(7)	+0.05 Å
C(2)	+0.01		
C(3)	-0.01		
C(4)	-0.01		
C(5)	+0.02		
C(6)	-0.01		

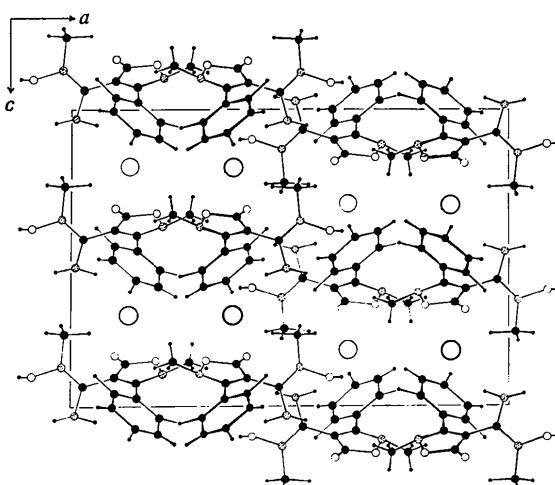
(c) Least-squares plane of the amidinic chain

$$1.172x + 10.656y + 0.183z - 3.818 = 0$$

N(2)	-0.06 Å	C(9)	+0.23 Å
C(10)	+0.05		
N(3)	+0.07		
O(2)	0.00		
C(11)	-0.06		

The proton of the hydrobromic acid has been found linked to the iminic nitrogen atom N(2).

In the amidinic chain, the distances C(10)—N(3) and C(10)—N(2) are equal; a resonance of the following type

Fig. 1. Projection of the structure along the *b* axis.

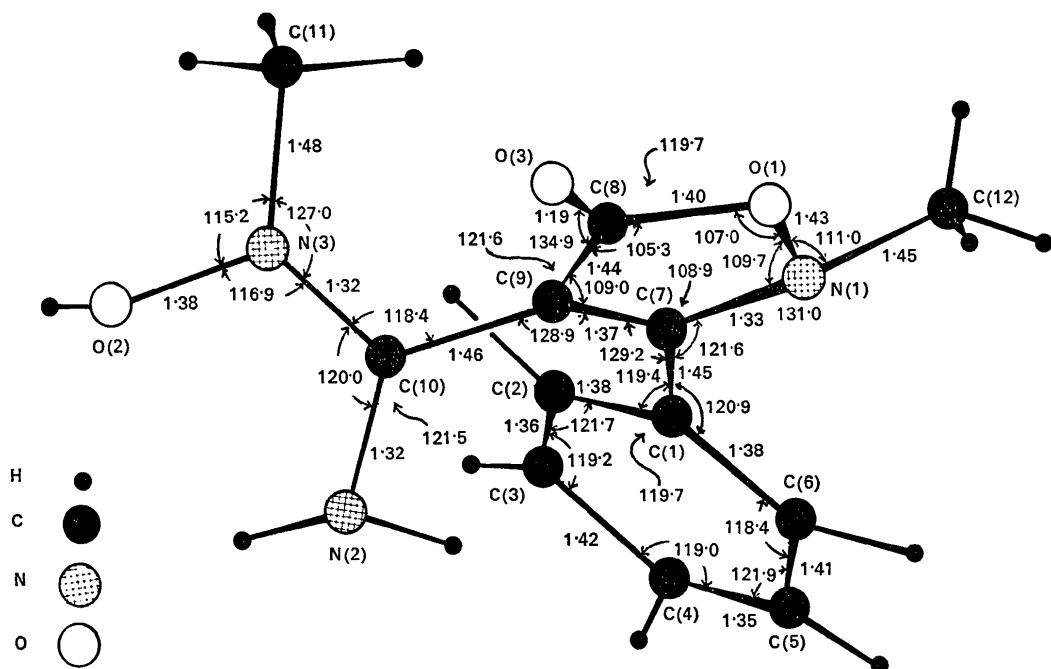
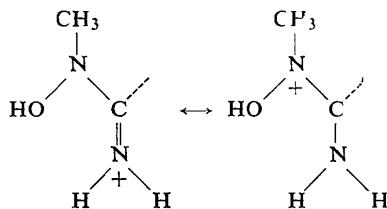


Fig. 2. Intramolecular bond distances and angles.



can account for this and for the approximate coplanarity of O(2), N(3), C(10), N(2) and C(9) (the equation of the least-squares plane – plane III – is given in Table 6, with the deviations of the atoms from the plane).

The C–H bond lengths have an average value of 1.02 Å. The mean N–H distance is 0.95 Å. Each bromide ion links two different molecules by hydrogen bonds to O(2) of one molecule and N(2) of another.

The distance Br···O(2) is 3.09, Br···N(2) 3.31 Å. The corresponding bromine–hydrogen distances are Br···HO(2) 2.05 Å and Br···H(1) N(2) 2.53 Å.

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