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The Crystal Structure of *m*-Nitrobenzoic Acid*

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Crystals of *m*-nitrobenzoic acid, $NO_2C_6H_4COOH$, are monoclinic, space group $P2_1/c$ with $a = 13{\cdot}22 \pm 0{\cdot}01$, $b = 10{\cdot}67 \pm 0{\cdot}01$, $c = 10{\cdot}37 \pm 0{\cdot}01$ Å; $\beta = 91{\cdot}2 \pm 0{\cdot}1°$; $\varrho_o = 1{\cdot}519$ g cm^{-3}; ϱ_c for $Z = 8$ is 1·517 g cm^{-3}. The structure was solved by direct methods with visually estimated data, and refined by the method of least squares to an R value of 0·118 for 2013 observed reflexions (0·105 omitting seven reflexions which appear to have been affected by extinction). The e.s.d.s in bond lengths not involving hydrogen atoms are 0·007–0·010 Å and those in bond angles are about 0·6°. The structure consists of dimeric units, each consisting of crystallographically independent molecules *A* and *B*; the OH···O hydrogen bonds linking molecules *A* and *B* are 2·630 (7) and 2·666 (7) Å long. The maximum difference between the observed lengths of the equivalent bonds (not involving hydrogen atoms) in the two molecules *A* and *B* is 0·011 Å (average scatter, 0·004 Å). The environments of molecules *A* and *B* are quite different; this difference in environment accounts for minor but significant differences in the structures of the two molecules as well as in their thermal vibration patterns.

Introduction

The structure analysis of *m*-nitrobenzoic acid was undertaken by Arora (1968) in this laboratory but the correct trial structure could not be determined at that time. We have now been able to solve the structure with the Sayre's-equation program written by Long (1965). This work is a continuation of our earlier studies on the structures of *p*- and *o*-nitrobenzoic acids (Sakore & Pant, 1966; Tavale & Pant, 1971; Sakore, Tavale & Pant, 1967; Tavale & Pant, in preparation).

Experimental

The crystals grown from aqueous alcohol are monoclinic, space group $P2_1/c$, with $a = 13{\cdot}22 \pm 0{\cdot}01$, $b = 10{\cdot}67 \pm 0{\cdot}01$, $c = 10{\cdot}37 \pm 0{\cdot}01$ Å; $\beta = 91{\cdot}2 \pm 0{\cdot}1°$; $\varrho_o = 1{\cdot}519$ g cm^{-3}; ϱ_c for $Z = 8$ is 1·517 g cm^{-3}; these data are in substantial agreement with those of Thakar, Kapadia & Prasad (1940).

Axial lengths were measured from high-angle reflexions on zero-layer Weissenberg photographs with the films mounted in the Straumanis arrangement; β was determined by the method of triangulation (Jeffery, 1971). Data were collected with unfiltered Cu radiation from the zero to ninth layer Weissenberg photographs about the *b* axis and from the zero to fourth layer photographs about the *c* axis; the crystals used for the two sets of photographs had the cross-sections $(0{\cdot}5 \times 1{\cdot}0)$ mm^2 and $(0{\cdot}8 \times 1{\cdot}2)$ mm^2 respectively. The data were processed in the usual way (Takwale & Pant, 1971); absorption was neglected.

Determination and refinement of the structure

Normalized values of the 128 structure factors having unitary values $\geq 0{\cdot}2$ (listed by Arora, 1968) were used in the Sayre's-equation program written by Long (1965); signs of 123 *F*'s were derived and were used to obtain a three-dimensional Fourier map. 23 of the 24 non-hydrogen atoms were located on the map; the position of the 24th atom (O of a nitro group) was fixed by stereochemistry. The trial structure was first refined isotropically with unit weights for all reflexions by the block-diagonal least-squares method; later, hydrogen atoms were included and anisotropic temperature factors were introduced, and the refinement continued with Cruickshank's weighting scheme. R decreased to 0·148. Since R was rather high in spite of fairly good data, the reflexions obtained from different photographs were rescaled from the F_c values. 131 strong low-angle reflexions were remeasured on the zero to fifth layer integrated Weissenberg photographs and scaled by means of the F_c's. The structure was refined for four more cycles with the revised F_o's included. The final R is 0·118 for 2013 observed reflexions; omission of seven strong low-angle reflexions for which $\Delta F > 30$ reduced R to 0·105. For all these reflexions $F_o < F_c$ and they appear to be affected by extinction. The final atomic and thermal parameters together with their e.s.d.s (obtained from the diagonal terms of the least-squares inverse matrices) are given in Table 1.

The intramolecular bond lengths and angles in the two crystallographically independent molecules *A* and *B* are listed in Table 2 together with their e.s.d.s obtained in the usual way assuming isotropic errors; the atoms of molecule *B* are indicated by primed letters.

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The equations of the various planes referred to the a', b, c orthogonal axes are given in Table 3 together with the angles between them and the deviations of the atoms from the different planes. The corrections to some of the bond lengths in the carboxylic and the nitro groups arising from large non-rigid-body vibrations were evaluated in the usual way (Takwale & Pant, 1971); details of the atomic thermal vibration ellipsoids have been omitted for brevity.

Description of the structure and discussion

The structure consists of dimeric units, each consisting of crystallographically independent molecules *A* and *B*; unlike many other crystalline carboxylic acid structures, the dimer in this structure is not symmetrical. The OH···O hydrogen bonds linking molecules *A* and *B* are 2·630 (7) and 2·666 (7) Å long. The environment of the two molecules forming the dimer is quite different and this difference in environment accounts for minor but significant differences in the structures of the two molecules as well as in their thermal vibration patterns.

The lengths of the equivalent bonds of the benzene rings in *A* and *B* agree within about 0·005 Å. In molecule *A*, the nitro group makes an angle of 21·7° with the aromatic plane while the corresponding angle in molecule *B* is 5·3°. The equivalent bond lengths in the nitro groups of molecules *A* and *B* agree within about 0·011 Å. The corresponding bond lengths in the carboxylic groups of *A* and *B* agree within about 0·006 Å; the carboxylic group of molecule *A* makes an angle of 4·5° with the attached aromatic ring while the corresponding angle for molecule *B* is 1·6°.

A comparison of the mean molecular dimensions (average for *A* and *B*) of *m*-nitrobenzoic acid with the molecular dimensions of *p*-nitrobenzoic acid (Tavale & Pant, 1971) suggests that the two structures are quite similar. In particular, the distribution of bond lengths in the aromatic ring with respect to the nitro group is similar in the two structures; the aromatic bonds parallel to the C–N bond are 1·384 and 1·382 Å long in *m*-nitrobenzoic acid, and 1·384 and 1·381 Å long in *p*-nitrobenzoic acid. The dimensions of the nitro group also agree in the two molecules. These results probably imply that in both molecules the nitro group

Table 1. *Final atomic and thermal parameters and their estimated standard deviations* (*in parentheses*)

Anisotropic thermal parameters are of the form $T=\exp\,[-(b_{11}h^2+b_{22}k^2+b_{33}l^2+2b_{12}hk+2b_{23}kl+2b_{13}hl)]$.

(*a*) Non-hydrogen atoms ($\times 10^4$)

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{23}	b_{13}
C(1)	2346 (5)	1622 (6)	3001 (5)	58 (4)	94 (6)	74 (5)	0 (4)	−8 (5)	3 (3)
C(2)	1683 (4)	637 (6)	2782 (6)	55 (4)	98 (6)	78 (5)	4 (4)	1 (5)	−3 (4)
C(3)	893 (4)	820 (6)	1901 (6)	45 (3)	108 (7)	82 (5)	−1 (4)	−20 (5)	4 (3)
C(4)	742 (5)	1925 (6)	1233 (7)	60 (4)	103 (7)	115 (7)	18 (4)	0 (6)	−17 (4)
C(5)	1423 (5)	2890 (6)	1433 (7)	68 (4)	100 (6)	102 (6)	10 (4)	2 (6)	−8 (4)
C(6)	2234 (5)	2738 (6)	2313 (6)	65 (4)	101 (7)	93 (6)	5 (4)	−12 (5)	−3 (4)
C(7)	3196 (4)	1456 (6)	3958 (6)	52 (3)	88 (6)	89 (5)	2 (4)	−16 (5)	−5 (4)
O(1)	3820 (4)	2373 (5)	4052 (5)	70 (3)	113 (5)	135 (5)	−18 (3)	19 (4)	−34 (3)
O(2)	3256 (3)	484 (4)	4629 (4)	63 (3)	97 (4)	107 (4)	−4 (3)	10 (4)	−19 (3)
O(3)	483 (4)	−1290 (5)	1911 (5)	75 (3)	106 (5)	152 (6)	−19 (3)	2 (5)	−5 (4)
O(4)	−645 (3)	7 (5)	1180 (4)	47 (2)	145 (6)	120 (5)	−8 (3)	−5 (4)	−9 (3)
N	199 (4)	−233 (5)	1655 (5)	54 (3)	111 (6)	87 (5)	−6 (3)	−6 (4)	1 (3)
C′(1)	5878 (4)	1262 (5)	7851 (5)	51 (3)	76 (5)	83 (5)	9 (3)	−1 (4)	−1 (3)
C′(2)	6546 (4)	2255 (6)	7965 (5)	61 (4)	80 (6)	81 (5)	−1 (4)	6 (5)	−4 (4)
C′(3)	7257 (4)	2236 (6)	8971 (6)	48 (3)	100 (6)	89 (6)	9 (4)	−11 (5)	−4 (3)
C′(4)	7326 (5)	1269 (7)	9860 (6)	61 (4)	121 (7)	82 (5)	19 (4)	0 (5)	−14 (4)
C′(5)	6640 (5)	295 (7)	9743 (7)	67 (4)	128 (8)	99 (6)	6 (5)	23 (6)	4 (4)
C′(6)	5920 (5)	277 (6)	8729 (6)	65 (4)	97 (6)	99 (6)	5 (4)	15 (5)	12 (4)
C′(7)	5136 (4)	1278 (6)	6752 (6)	50 (3)	86 (6)	97 (6)	2 (4)	−1 (5)	−1 (3)
O′(1)	4541 (3)	328 (5)	6639 (5)	63 (3)	113 (5)	131 (5)	−17 (3)	1 (4)	−26 (3)
O′(2)	5131 (4)	2171 (5)	5984 (5)	83 (3)	120 (5)	117 (5)	−18 (3)	30 (4)	−36 (3)
O′(3)	7972 (4)	4073 (5)	8241 (6)	105 (4)	124 (6)	143 (6)	−45 (4)	−3 (5)	−12 (4)
O′(4)	8557 (4)	3304 (5)	10022 (6)	86 (4)	145 (6)	162 (7)	3 (4)	−43 (5)	−58 (4)
N′	7982 (4)	3284 (5)	9089 (5)	69 (4)	104 (6)	107 (6)	−1 (4)	−29 (5)	−11 (4)

(*b*) Hydrogen atoms ($\times 10^3$)

	x	y	z
H(1)	170 (5)	−14 (7)	316 (7)
H(2)	16 (6)	194 (8)	73 (8)
H(3)	127 (6)	388 (7)	105 (8)
H(4)	260 (6)	342 (8)	255 (8)
H(5)	436 (6)	225 (8)	456 (9)
H′(1)	666 (6)	290 (7)	756 (7)
H′(2)	781 (6)	117 (8)	1052 (7)
H′(3)	659 (6)	−44 (8)	1040 (8)
H′(4)	542 (6)	−35 (8)	864 (8)
H′(5)	396 (6)	46 (8)	640 (8)

Table 2. *Intramolecular bond lengths and angles and their standard deviations* (*in parentheses*)

(*a*) Bond lengths

	Molecule *A*			Molecule *B*	
	Uncorrected	Corrected		Uncorrected	Corrected
C(1)–C(2)	1·384 (9) Å		C′(1)–C′(2)	1·383 (8) Å	
C(2)–C(3)	1·387 (9)		C′(2)–C′(3)	1·390 (8)	
C(3)–C(4)	1·380 (9)		C′(3)–C′(4)	1·385 (9)	
C(4)–C(5)	1·381 (10)		C′(4)–C′(5)	1·383 (10)	
C(5)–C(6)	1·403 (9)		C′(5)–C′(6)	1·404 (10)	
C(6)–C(1)	1·394 (9)		C′(6)–C′(1)	1·391 (8)	
C(7)–C(1)	1·494 (9)		C′(7)–C′(1)	1·488 (8)	
C(7)–O(1)	1·282 (8)	1·297 Å	C′(7)–O′(1)	1·287 (8)	1·298 Å
C(7)–O(2)	1·251 (7)	1·260	C′(7)–O′(2)	1·242 (8)	1·258
C(3)–N	1·470 (8)	1·474	C′(3)–N′	1·476 (8)	1·485
N—–O(3)	1·216 (8)	1·232	N′—–O′(3)	1·217 (8)	1·233
N—–O(4)	1·237 (7)	1·250	N′—–O′(4)	1·218 (8)	1·243
C(2)–H(1)	0·92 (7)		C′(2)–H′(1)	0·82 (8)	
C(4)–H(2)	0·92 (8)		C′(4)–H′(2)	0·94 (8)	
C(5)–H(3)	1·14 (8)		C′(5)–H′(3)	1·04 (8)	
C(6)–H(4)	0·90 (8)		C′(6)–H′(4)	0·94 (8)	
O(1)–H(5)	0·89 (8)		O′(1)–H′(5)	0·82 (8)	

(*b*) Bond angles

Molecule *A*		Molecule *B*	
C(6)–C(1)–C(2)	120·3 (6)°	C′(6)–C′(1)–C′(2)	120·4 (5)°
C(1)–C(2)–C(3)	117·8 (6)	C′(1)–C′(2)–C′(3)	118·3 (5)
C(2)–C(3)–C(4)	123·4 (6)	C′(2)–C′(3)–C′(4)	123·0 (6)
C(3)–C(4)–C(5)	118·3 (6)	C′(3)–C′(4)–C′(5)	117·9 (6)
C(4)–C(5)–C(6)	119·9 (6)	C′(4)–C′(5)–C′(6)	120·6 (6)
C(5)–C(6)–C(1)	120·2 (6)	C′(5)–C′(6)–C′(1)	119·8 (6)
C(1)–C(7)–O(1)	115·7 (5)	C′(1)–C′(7)–O′(1)	116·9 (5)
C(1)–C(7)–O(2)	120·4 (5)	C′(1)–C′(7)–O′(2)	119·6 (5)
O(1)–C(7)–O(2)	123·9 (6)	O′(1)–C′(7)–O′(2)	123·4 (6)
C(3)–N—–O(3)	118·8 (5)	C′(3)–N′—–O′(3)	117·8 (5)
C(3)–N—–O(4)	117·7 (5)	C′(3)–N′—–O′(4)	118·2 (5)
O(3)–N—–O(4)	123·4 (5)	O′(3)–N′—–O′(4)	124·0 (6)
C(2)–C(1)–C(7)	118·9 (5)	C′(2)–C′(1)–C′(7)	117·8 (5)
C(6)–C(1)–C(7)	120·8 (6)	C′(6)–C′(1)–C′(7)	121·8 (5)
C(2)–C(3)–N	117·7 (5)	C′(2)–C′(3)–N′	118·6 (5)
C(4)–C(3)–N	118·9 (5)	C′(4)–C′(3)–N′	118·4 (5)

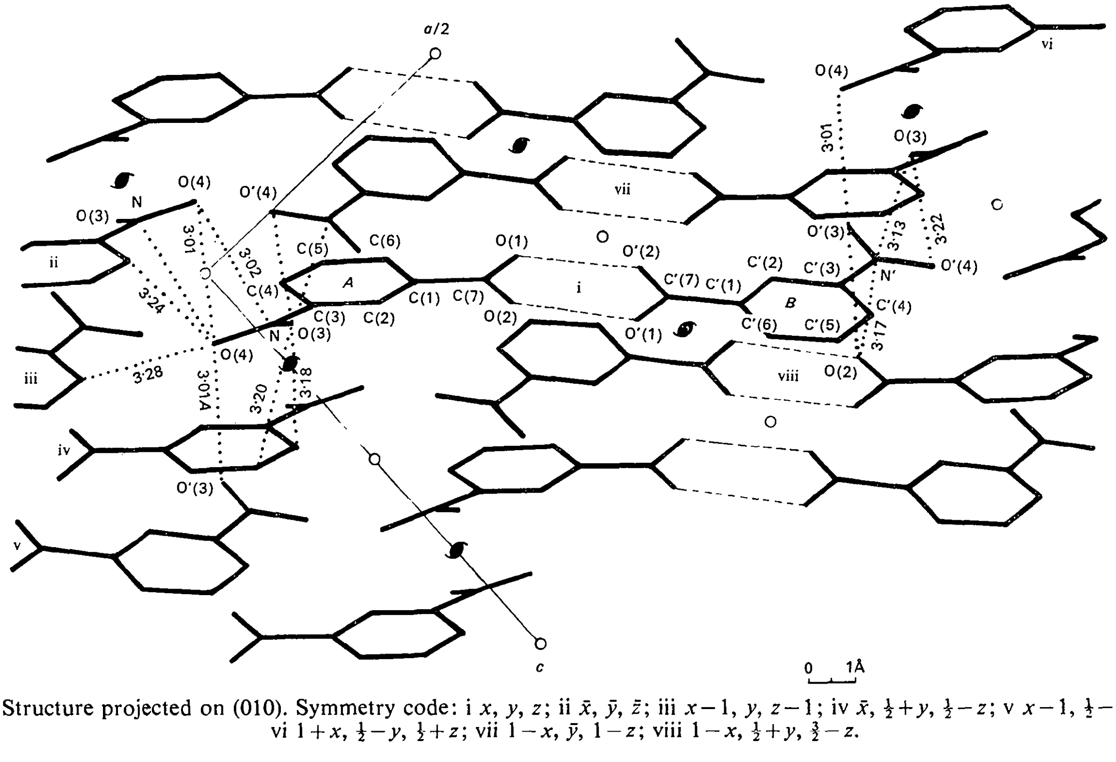

Fig. 1. Structure projected on (010). Symmetry code: i x, y, z; ii $\bar{x}, \bar{y}, \bar{z}$; iii $x-1, y, z-1$; iv $\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z$; v $x-1, \frac{1}{2}-y, z-\frac{1}{2}$; vi $1+x, \frac{1}{2}-y, \frac{1}{2}+z$; vii $1-x, \bar{y}, 1-z$; viii $1-x, \frac{1}{2}+y, \frac{3}{2}-z$.

interacts with the aromatic ring to a much greater extent than does the carboxylic group. It may be mentioned that there is no apparent similarity in the structures of *m*-nitrobenzoic acid and *o*-nitrobenzoic acid (Tavale & Pant, in preparation) presumably because of large steric interactions in the latter molecule.

The planarity of the benzene ring of molecule *A* is disturbed to a greater extent than that of molecule *B*; the mean deviation of an atom of the ring from the least-squares plane is 0·009 Å for molecule *A* and 0·005 Å for molecule *B*. However, the more striking difference between the two molecules is the much greater r.m.s. amplitudes of vibration of the atoms of the nitro group of molecule *B* compared with those of molecule *A*. The reason is clear from the crystal structure shown in the (010) projection in Fig. 1. The nitro group of molecule *A* has many more and shorter intermolecular contacts than the nitro group of molecule *B*; therefore the vibrations of the atoms of the former are more restricted.

The nitrogen atom N of molecule *A* deviates 0·051 Å from the least-squares plane of the attached benzene ring while the corresponding deviation for N′ of molecule *B* is only 0·011 Å (Table 3). The greater deviation of N is due to the greater asymmetry of its environment. N′ has two short contacts (< 3·3 Å) of 3·17 and 3·13 Å with O(2viii) and O(3vii) respectively whereas N has only one contact of 3·02 Å with O(4ii) (Fig. 1). The N–O(4ii) contact makes an angle of 54° with the benzene plane and 88° with the C(3)–N bond. It is worth mentioning that the atom N deviates from the aromatic plane towards the O(4ii) atom; it is probably

Table 3. *Equations of planes*

(*a*) Molecule *A*

(1) Benzene ring

$$-0{\cdot}5708X'+0{\cdot}3504Y+0{\cdot}7426Z'=1{\cdot}0837\ \text{Å}$$

(2) Carboxylic group

$$-0{\cdot}5582X'+0{\cdot}4236Y+0{\cdot}7134Z'=1{\cdot}1739$$

(3) Nitro group

$$-0{\cdot}3626X'+0{\cdot}0962Y+0{\cdot}9270Z'=1{\cdot}4607$$

(*b*) Molecule *B*

(4) Benzene ring

$$-0{\cdot}6411X'+0{\cdot}4837Y+0{\cdot}5959Z'=0{\cdot}4276\ \text{Å}$$

(5) Carboxylic group

$$-0{\cdot}6653X'+0{\cdot}4691Y+0{\cdot}5808Z'=0{\cdot}1000$$

(6) Nitro group

$$0{\cdot}6702X'-0{\cdot}5293Y-0{\cdot}5203Z'=0{\cdot}4296$$

(7) Plane through O(1), O(2), O′(1) and O′(2)

$$-0{\cdot}6573X'+0{\cdot}4504Y+0{\cdot}6042Z'=0{\cdot}2734$$

Dihedral angles between planes (°)

	Molecule *A*			Molecule *B*	
	(2)	(3)		(5)	(6)
(1)	4·5	21·7	(4)	1·6	5·3

Deviations of atoms from planes (Å)

	Molecule *A* Planes			
	(1)	(2)	(3)	(7)
C(1)	0·015	0·002		
C(2)	−0·008			
C(3)	−0·005		0·000	
C(4)	0·010			
C(5)	−0·003			
C(6)	−0·010			
C(7)	0·032	−0·008		0·077
O(1)	−0·037	0·003		0·024
O(2)	0·138	0·003		−0·023
O(3)	−0·469		0·000	
O(4)	0·327		0·000	
N	−0·051		0·006	
H(1)	−0·015			
H(2)	0·081			
H(3)	0·189			
H(4)	0·147			
H(5)	−0·109	−0·085		

	Molecule *B* Planes			
	(4)	(5)	(6)	(7)
C′(1)	−0·002	−0·002		
C′(2)	0·004			
C′(3)	0·001		0·000	
C′(4)	−0·008			
C′(5)	0·010			
C′(6)	−0·004			
C′(7)	−0·032	0·008		0·024
O′(1)	−0·078	−0·003		0·023
O′(2)	−0·041	−0·003		−0·024
O′(3)	−0·119		0·000	
O′(4)	0·079		0·000	
N′	−0·011		−0·003	
H′(1)	−0·007			
H′(2)	−0·073			
H′(3)	0·082			
H′(4)	0·047			
H′(5)	0·346	0·442		

reasonable to conclude that there is electrostatic interaction between the oppositely charged N and O(4) atoms.

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Structure Cristalline de la *cis*-Dibromo-2,4 Ditertiobutyl-2,4 Cyclobutanone

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cis-2,4-Dibromo-2,4-di-t-butylcyclobutanone crystallizes in the triclinic space group $P\bar{1}$ with unit-cell dimensions $a=6{\cdot}16$, $b=9{\cdot}80$, $c=13{\cdot}05$ Å, $\alpha=106{\cdot}20$, $\beta=104$ and $\gamma=103{\cdot}20°$. The atomic positions of all the atoms have been refined with anisotropic temperature factors for the bromide atoms and isotropic temperature factors for the carbon and oxygen atoms, to an R value of 0·11 for 1359 photographic data. The cyclobutanone ring is puckered. Two short Br · · · Br intermolecular distances of 3·55 Å are observed.

Introduction

Les cyclobutanones ont été très étudiées ces dernières années dans le laboratoire de M Conia. Les nombreux résultats obtenus dans l'analyse structurale de cyclobutanones diversement substituées ont mis en évidence la non-planéité et la mobilité conformationnelle du cycle (Revue: Conia, 1966; Conia & Salaün, 1965).

Nous décrirons dans ce mémoire la structure cristalline de la *cis*-dibromo-2,4 ditertiobutyl-2,4 cyclobutanone.

Partie expérimentale

Les cristaux se présentent sous la forme de petites plaquettes donnant une extinction oblique en lumière polarisée. Ils appartiennent au système triclinique. Les paramètres de la maille ont été déterminés à partir de clichés de cristal tournant et de Weissenberg, avec étalonnage par fil d'aluminium.

Les principales données cristallographiques sont rassemblées dans le Tableau 1.

Le groupe spatial $P\bar{1}$ a été choisi et, par la suite, ce choix a été confirmé lors de la résolution de la structure.

Nous avons enregistré les taches de diffraction par la méthode photographique des films superposés, à l'aide d'un goniomètre de Weissenberg utilisé en équi-inclinaison, avec la radiation $K\alpha$ du cuivre. Le composé se sublimant à l'air, nous avons employé un cristal différent pour chaque strate. Chacune des réflexions a été mesurée deux fois: sur le haut et sur le bas des films, avec un microdensitomètre manuel. La moyenne harmonique de ces mesures a fourni les intensités qui ont été corrigées des facteurs de Lorentz–polarisation et du dédoublement causé par la structure fine $K\alpha_1\alpha_2$ de la radiation utilisée.

Tableau 1. *Données cristallographiques*

Système triclinique	
Groupe spatial	$P\bar{1}$
a	6,16 ± 0,01 Å
b	9,80 ± 0,02
c	13,05 ± 0,01
α	106,20 ± 0,20°
β	104 ± 0,20
γ	103,20 ± 0,20
V	694 Å^3
$F(000)$	300 e
Radiation utilisée	1,5418 Å
Nombre de réflexions observées	1359

Résolution et affinement de la structure

La structure a été résolue (Riche, 1966) sur les projections $0yz$ et $xy0$, par la méthode de l'atome lourd.

Les paramètres atomiques ont été affinés par la méthode des moindres carrés (matrice complète). La quan-

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