

The Crystal and Molecular Structure of 2-Methoxyisonitrosoacetanilide

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The crystal and molecular structures of 2-methoxyisonitrosoacetanilide have been solved by direct methods and refined by block-diagonal least-squares methods with diffractometer-collected intensities. The crystals are orthorhombic, space group $P2_12_12_1$, with unit-cell dimensions $a=16.384$ (5), $b=24.613$ (10), $c=4.669$ (2) Å, $Z=8$. The final R value is 0.045 for 1737 observed reflexions; all bond distances and angles are in good agreement with expected values. The two molecules in the asymmetric unit have slightly different conformations. The cohesion of the crystal is produced by van der Waals interactions and hydrogen bonds.

Introduction

Four alkoxy derivatives of isonitrosoacetanilide, 2-methoxy, 4-methoxy, 2-ethoxy and 4-ethoxyisonitrosoacetanilide, have in turn been obtained from *o* and *p*-anisidine, and *o* and *p*-phenetidine respectively. The structures of the last three have been published (Briansó, Miravittles, Plana & Font-Altaba, 1974; Briansó, Miravittles, Plana & Font-Altaba, 1973; Plana, Miravittles, Briansó & Font-Altaba, 1973); 2-methoxyisonitrosoacetanilide was the only one remaining to be resolved and its structure is now described.

The systematic structural study of all the derivatives of isonitrosoacetanilide was carried out in our laboratory. Among these, the structures of the following have been resolved: isonitrosoacetanilide (Briansó, Miravittles, Font-Altaba, Declercq & Germain, 1973); 3-methyl-*N*-ethylisonitrosoacetanilide (Plana, Miravittles, Briansó & Font-Altaba, 1974); 2-chloro (Miravittles, Plana, Briansó & Font-Altaba, 1974) and *N*-ethanolisonitrosoacetanilide, the latter being the cyclic isomer *N*-ethanol- β -isatoxime (Plana, Briansó, Miravittles & Font-Altaba, 1973). The introduction of alkoxy substituents on the benzene ring has no influence on the reactive group. The study of some of the reactions of this group of four derivatives has enabled the establishment of a series of colorimetric methods which allow the identification of the Pd and Co ions (Buscarons & Mena, 1963).

At the same time, a study of the structure of a large number of derivatives with different substituents on the benzene ring allowed us to study the symmetry as a function of these substituents, the planarity of the

benzene ring and the effect of these substituents on the physical and chemical properties of the compounds.

Experimental

2-Methoxyisonitrosoacetanilide is difficult to crystallize and we have been able to obtain only two crystals of acceptable quality. The first was obtained by evaporation of an ethanol solution of the crystalline powder at room temperature, where the majority of crystals obtained were of a poor quality and it was only possible to isolate one of them. With this crystal (1) the cell parameters were determined from Weissenberg diagrams and the intensities were measured with an automatic four-circle diffractometer. The intensities of 1195 reflexions were obtained, of which only 890 were considered as observed. The attempts made to obtain the structure from this data were totally fruitless. Crystal (2) was obtained by controlled slow evaporation from an ethanol solution at constant temperature over four months, in order to avoid the effects of thermal shocks and consequent formation of imperfections. The crystal obtained has a prismatic habit, elongated in the *c* direction, and a drab brown colour. It measured $0.2 \times 0.2 \times 0.5$ mm. Cell parameters were obtained by least-squares refinement of general reflexions measured on a diffractometer.

Crystal data

$C_9H_{10}N_2O_3$, orthorhombic, space group $P2_12_12_1$; $a=16.384$ (5), $b=24.613$ (10), $c=4.669$ (2) Å, $V=1.883$ Å³, $D_c=1.368$ g cm⁻³, $Z=8$, $\lambda(\text{Cu } K\alpha)=1.54178$ Å.

The intensity data were collected on a Hilger &

Watts four-circle diffractometer, and all reflexions having $2\theta < 70^\circ$ were collected. The intensities of 1878 independent reflexions were measured, and 1737, with $I > 2\sigma(I)$, were used in the analysis. Lorentz and polarization corrections were applied to the raw data and relative structure amplitudes derived. No corrections were made for absorption or extinction.

Structure solution and refinement

This structure could not be solved by a straightforward application of *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974). Eventually it was

solved by applying a substantialization technique with *MULTAN* (Woolfson 1954; Germain & Woolfson, 1968). Substantialization was carried out with seven special reflexions in the starting set, each of which could take one of two values, so that only 16 combinations of phases had to be tested. One of them revealed an eight-atom fragment from which the entire structure was developed by tangent-formula recycling (Karle, 1968). A detailed analysis of the solving process for this structure is given by Lessinger (1976). After five cycles of block-diagonal least-squares refinement (*SFLS*, Ahmed, 1966) with individual isotropic thermal parameters, R was 0.062.

In the subsequent step, a Fourier difference map gave coordinates for all the H atoms. The atomic parameters of all atoms were refined by four cycles to $R = 0.045$ with all observed reflexions. An overall isotropic thermal parameter of $B = 4.281 \text{ \AA}^2$ was assigned to the H atoms. The weighting scheme used was that given by Cruickshank (1965); $w = 1/[P_1 + |F_o|^2 + P_2|F_o|^2]$ with $P_1 = 2F_{\min}$, $P_2 = 2/F_{\max}$. The atomic scattering factors used were taken from *International Tables for X-ray Crystallography* (1962). Tables 1 and 2* give the final atomic coordinates and thermal parameters and their estimated standard deviations.

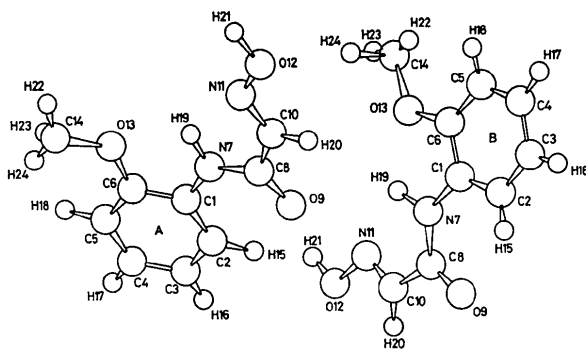


Fig. 1. Atomic numbering of two independent molecules viewed along the c axis.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31783 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final structure parameters for the non-hydrogen atoms*

Standard deviations are in parentheses. All values have been multiplied by 10^5 . The temperature factor is of the form: $\exp [-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)]$.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
C(1) <i>A</i>	92105 (18)	59762 (13)	88513 (72)	276 (12)	164 (5)	3960 (148)	-52 (50)	-249 (74)	-13 (13)
C(2) <i>A</i>	98491 (21)	62360 (14)	74528 (84)	327 (14)	205 (6)	4690 (179)	141 (61)	-216 (88)	-48 (16)
C(3) <i>A</i>	103028 (21)	59596 (17)	54061 (89)	307 (14)	286 (8)	4976 (184)	287 (71)	235 (95)	55 (18)
C(4) <i>A</i>	101199 (23)	54238 (17)	47628 (93)	429 (16)	277 (8)	4651 (188)	-40 (73)	110 (99)	204 (19)
C(5) <i>A</i>	94843 (23)	51563 (14)	61466 (91)	427 (15)	192 (6)	5394 (196)	-201 (64)	-368 (100)	119 (16)
C(6) <i>A</i>	90293 (20)	54346 (13)	81602 (80)	319 (13)	159 (5)	4538 (160)	-21 (55)	-405 (81)	22 (13)
N(7) <i>A</i>	87093 (16)	62217 (10)	109444 (65)	281 (10)	142 (4)	4666 (142)	-90 (42)	45 (68)	-61 (10)
C(8) <i>A</i>	87886 (20)	67196 (12)	121076 (75)	331 (13)	153 (5)	4310 (161)	36 (52)	-312 (83)	-49 (14)
O(9) <i>A</i>	93160 (17)	70485 (10)	113843 (64)	549 (12)	185 (4)	5785 (144)	-173 (46)	721 (81)	-290 (13)
C(10) <i>A</i>	82098 (20)	68627 (13)	143834 (84)	368 (14)	160 (5)	4850 (178)	-228 (57)	218 (95)	-36 (14)
N(11) <i>A</i>	76129 (16)	65507 (11)	149960 (64)	317 (11)	173 (5)	4660 (145)	-194 (49)	-23 (73)	43 (12)
O(12) <i>A</i>	71623 (15)	67519 (11)	172758 (65)	381 (10)	229 (5)	6589 (159)	-587 (51)	788 (74)	-0 (12)
O(13) <i>A</i>	83805 (15)	52205 (8)	96315 (62)	412 (10)	140 (3)	6280 (146)	-252 (45)	415 (75)	-91 (10)
C(14) <i>A</i>	82144 (27)	46548 (13)	93029 (109)	585 (20)	127 (5)	7108 (263)	-276 (69)	23 (141)	-48 (17)
C(1) <i>B</i>	89160 (21)	87699 (13)	21717 (78)	346 (14)	164 (5)	4311 (163)	124 (53)	140 (84)	-16 (14)
C(2) <i>B</i>	91709 (22)	91736 (15)	40115 (90)	440 (16)	190 (6)	4937 (191)	-22 (64)	-199 (101)	-24 (16)
C(3) <i>B</i>	85978 (25)	94911 (15)	54165 (97)	552 (18)	197 (6)	5456 (210)	-213 (68)	385 (117)	17 (19)
C(4) <i>B</i>	77786 (25)	94020 (16)	49866 (109)	534 (18)	208 (7)	6627 (263)	-154 (81)	830 (126)	72 (19)
C(5) <i>B</i>	75150 (24)	89944 (17)	31794 (113)	378 (16)	242 (8)	7304 (259)	-47 (87)	472 (122)	21 (19)
C(6) <i>B</i>	80847 (22)	86706 (14)	17948 (90)	372 (15)	186 (6)	5475 (182)	-101 (63)	156 (94)	-17 (15)
N(7) <i>B</i>	94413 (16)	84347 (11)	5140 (68)	285 (10)	190 (5)	4888 (140)	-162 (49)	-292 (74)	-6 (12)
C(8) <i>B</i>	102575 (20)	84545 (12)	2484 (78)	327 (13)	143 (5)	4556 (163)	220 (52)	-197 (84)	-41 (13)
O(9) <i>B</i>	107005 (14)	87611 (9)	16254 (67)	317 (9)	194 (4)	6678 (151)	-368 (48)	-295 (73)	-58 (10)
C(10) <i>B</i>	106052 (20)	80721 (13)	-18772 (81)	302 (12)	173 (5)	4981 (171)	-32 (58)	-77 (88)	-40 (14)
N(11) <i>B</i>	101175 (18)	78103 (10)	-35137 (68)	407 (12)	159 (4)	4417 (140)	105 (46)	258 (76)	-46 (12)
O(12) <i>B</i>	105179 (15)	74776 (9)	-54275 (63)	426 (10)	185 (4)	5738 (146)	-332 (46)	452 (74)	-73 (11)
O(13) <i>B</i>	78921 (15)	82536 (11)	-475 (75)	328 (9)	262 (5)	8292 (200)	-859 (64)	-287 (82)	-60 (12)
C(14) <i>B</i>	70559 (24)	81158 (20)	-3844 (140)	374 (15)	325 (10)	10153 (389)	-1297 (118)	-65 (149)	-109 (21)

Table 2. Final structure parameters of the hydrogen atoms

Standard deviations are in parentheses. All values have been multiplied by 10^4 .

	x	y	z
H(15)A	9990 (27)	6644 (15)	7290 (114)
H(16)A	10734 (24)	6150 (15)	4282 (114)
H(17)A	10446 (26)	5203 (16)	3190 (110)
H(18)A	9351 (25)	4762 (16)	5628 (107)
H(19)A	8295 (25)	6004 (15)	11822 (118)
H(20)A	8336 (26)	7242 (15)	15386 (109)
H(21)A	6664 (26)	6506 (16)	17682 (109)
H(22)A	7686 (24)	4603 (17)	10427 (112)
H(23)A	8018 (26)	4664 (16)	7578 (102)
H(24)A	8652 (25)	4445 (16)	8045 (104)
H(15)B	9718 (24)	9220 (15)	4304 (109)
H(16)B	8740 (25)	9776 (16)	6648 (109)
H(17)B	7374 (25)	9611 (16)	5637 (111)
H(18)B	6896 (25)	8888 (15)	3086 (106)
H(19)B	9180 (25)	8137 (17)	-776 (109)
H(20)B	11216 (26)	8051 (16)	-1976 (103)
H(21)B	10046 (26)	7307 (16)	-6641 (112)
H(22)B	6762 (25)	8206 (15)	1527 (112)
H(23)B	6827 (26)	8346 (16)	-2084 (104)
H(24)B	7028 (25)	7703 (16)	-838 (108)

Discussion

The asymmetric unit consists of two 2-methoxyisonitrosoacetanilide molecules, *A* and *B*. Fig. 1 shows the molecules with the atomic numbering. The angles and distances are given in Tables 3 and 4, so that the equivalent distances and angles for the two molecules can be compared. The two molecules are practically identical. Their dimensions agree closely with those of the

other resolved structures in this series. It is worth noting that the angle C(1)–N(7)–C(8) of molecule *B* is 129° and C(1)–N(7)–C(8) of molecule *A* is 128° , these values being greater than expected due to the strain produced by the position of the atoms O(9) *B* and O(9) *A* respectively, the adjoining angles also being slightly affected.

Table 3. Intramolecular distances

Molecule A		Molecule B	
C(1)–C(2)	1.389 (4) Å	C(1)–C(2)	1.378 (5) Å
C(2)–C(3)	1.388 (5)	C(2)–C(3)	1.386 (5)
C(3)–C(4)	1.384 (5)	C(3)–C(4)	1.374 (5)
C(4)–C(5)	1.391 (5)	C(4)–C(5)	1.380 (6)
C(5)–C(6)	1.381 (5)	C(5)–C(6)	1.387 (5)
C(6)–C(1)	1.402 (4)	C(6)–C(1)	1.395 (4)
C(6)–O(13)	1.371 (4)	C(6)–O(13)	1.375 (4)
O(13)–C(14)	1.426 (4)	O(13)–C(14)	1.420 (4)
N(7)–C(1)	1.412 (4)	N(7)–C(1)	1.421 (4)
C(8)–N(7)	1.346 (4)	C(8)–N(7)	1.344 (4)
C(8)–O(9)	1.231 (4)	C(8)–O(9)	1.228 (4)
C(10)–C(8)	1.467 (4)	C(10)–C(8)	1.481 (4)
N(11)–C(10)	1.275 (4)	N(11)–C(10)	1.279 (4)
O(12)–N(11)	1.386 (4)	O(12)–N(11)	1.378 (4)
C(2)–H(15)	1.03 (4)	C(2)–H(15)	0.91 (4)
C(3)–H(16)	0.99 (4)	C(3)–H(16)	0.94 (4)
C(4)–H(17)	1.05 (4)	C(4)–H(17)	0.89 (4)
C(5)–H(18)	1.02 (4)	C(5)–H(18)	1.04 (4)
N(7)–H(19)	0.95 (4)	N(7)–H(19)	1.04 (4)
C(10)–H(20)	1.06 (4)	C(10)–H(20)	1.00 (4)
O(12)–H(21)	1.03 (4)	O(12)–H(21)	1.04 (4)
C(14)–H(22)	1.02 (4)	C(14)–H(22)	1.04 (5)
C(14)–H(23)	0.86 (4)	C(14)–H(23)	1.04 (4)
C(14)–H(24)	1.06 (4)	C(14)–H(24)	1.03 (4)

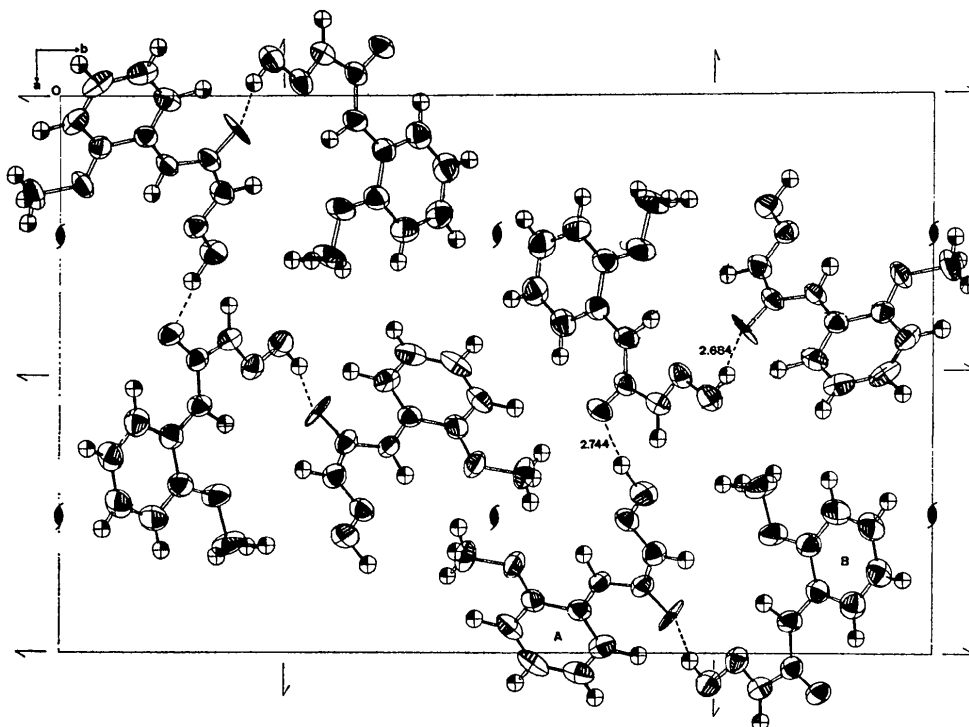


Fig. 2. Arrangement of molecules in the crystal viewed along the *c* axis. Hydrogen bonds are shown.

Table 4. *Intramolecular angles*

Molecule A		Molecule B	
N(7)—C(1)—C(2)	124.5 (1)°	N(7)—C(1)—C(2)	125.0 (2)°
N(7)—C(1)—C(6)	116.2 (1)	N(7)—C(1)—C(6)	114.9 (1)
C(6)—C(1)—C(2)	119.2 (1)	C(6)—C(1)—C(2)	120.0 (1)
C(1)—C(2)—C(3)	120.0 (2)	C(1)—C(2)—C(3)	119.7 (2)
C(2)—C(3)—C(4)	119.9 (2)	C(2)—C(3)—C(4)	120.1 (2)
C(3)—C(4)—C(5)	120.7 (2)	C(3)—C(4)—C(5)	120.7 (2)
C(4)—C(5)—C(6)	119.0 (2)	C(4)—C(5)—C(6)	119.4 (2)
C(5)—C(6)—C(1)	120.8 (1)	C(5)—C(6)—C(1)	119.8 (1)
C(5)—C(6)—O(13)	124.7 (2)	C(5)—C(6)—O(13)	124.4 (2)
C(1)—C(6)—O(13)	114.4 (2)	C(1)—C(6)—O(13)	115.7 (2)
C(8)—N(7)—C(1)	127.7 (1)	C(8)—N(7)—C(1)	129.2 (1)
C(10)—C(8)—N(7)	116.6 (1)	C(10)—C(8)—N(7)	114.9 (1)
O(9)—C(8)—N(7)	123.7 (2)	O(9)—C(8)—N(7)	124.2 (2)
C(10)—C(8)—O(9)	119.6 (2)	C(10)—C(8)—O(9)	120.9 (2)
N(11)—C(10)—C(8)	120.8 (2)	N(11)—C(10)—C(8)	118.6 (2)
O(12)—N(11)—C(10)	111.4 (1)	O(12)—N(11)—C(10)	112.9 (1)
C(6)—O(13)—C(14)	117.9 (1)	C(6)—O(13)—C(14)	117.9 (2)

In Table 5 the three mean planes calculated for each molecule have been given with their respective equations, and with the distances of the atoms from the plane and their standard deviations. The distances of the atoms of the benzene ring from the mean plane of the benzene are smaller in molecule A than in molecule B. In addition, the χ^2 value for the mean plane of the benzene ring of molecule A is 5.17 and for molecule B is 28.15, which shows that the benzene ring for molecule A is more nearly planar.

The most important torsion angles are given in Table 6; we can say that the distortions from coplanarity are small, and the largest ones correspond to N(11)A—C(10)A—C(8)A—N(7)A and N(11)B—C(10)B—C(8)B—N(7)B.

Fig. 2 shows a *c*-axis projection of the unit-cell constants with the anisotropic thermal parameters, drawn with the ORTEP program of Johnson (1965). The symmetry elements for each group and the hydrogen-bond links between the molecules A and B have been added. These hydrogen bonds are between the atoms O(12)B—H(21)B...O(9)A and O(12)A—H(21)A...O(9)B (Table 7). The cohesion of the crystal is pro-

Table 5. *Least-squares planes, atom deviations ($\times 10^3$ Å) and dihedral angles between planes*

	Molecule A				Molecule B				
Benzene ring:	0.6308x - 0.3069y + 0.7127z = 7.9504				-0.0020x - 0.6439y + 0.7651z = -13.1376				
	C(1)	2 (3)	C(4)	-1 (4)	C(1)	-12 (3)	C(4)	-4 (5)	
	C(2)	1 (3)	C(5)	4 (3)	C(2)	4 (4)	C(5)	-4 (5)	
	C(3)	-2 (3)	C(6)	-5 (3)	C(3)	4 (4)	C(6)	12 (4)	
		N(7)	-4 (3)			N(7)	-75 (3)		
Isonitrosoacetyl group:	0.6071x - 0.3875y + 0.6937z = 6.2523				0.0862x - 0.7283y + 0.6798z = -13.6358				
	N(7)	23 (2)	C(10)	29 (3)	N(7)	14 (3)	C(10)	69 (3)	
	C(8)	4 (3)	N(11)	-68 (2)	C(8)	10 (3)	N(11)	-49 (3)	
	O(9)	-18 (2)	O(12)	30 (2)	O(9)	-40 (3)	O(12)	-4 (2)	
Methoxy group:	0.6004x - 0.2027y + 0.7736z = 9.1193				0.0756x - 0.6542y + 0.7525z = -12.3236				
	Molecule A				Molecule B				
Benzene ring-isonitrosoacetyl group	5°				Benzene ring-isonitrosoacetyl group				8°
Benzene ring-methoxy group	7				Benzene ring-methoxy group				4
					Benzene ring (molecule A)-benzene ring (molecule B)				42

Table 6. *Torsion angles*

Molecule A		Molecule B	
C(8)N(7)C(1)C(6)	175°	C(8)N(7)C(1)C(6)	175°
C(14)O(13)C(6)C(1)	175	C(14)O(13)C(6)C(1)	176
O(12)N(11)C(10)C(8)	177	O(12)N(11)C(10)C(8)	179
C(1)N(7)C(8)C(10)	176	C(1)N(7)C(8)C(10)	175
O(13)C(6)C(1)N(7)	0	O(13)C(6)C(1)N(7)	2
N(11)C(10)C(8)N(7)	7	N(11)C(10)C(8)N(7)	9

Table 7. *Hydrogen-bond distances and angles*

O(12 ⁱ)B—H(21 ⁱ)B...O(9 ⁱⁱ)A	2.684 (3) Å
O(12 ⁱ)A—H(21 ⁱ)A...O(9 ⁱⁱⁱ)B	2.744 (3)
H(21 ⁱ)B—O(9 ⁱⁱ)A	1.63 (4)
H(21 ⁱ)A—O(9 ⁱⁱⁱ)B	1.73 (5)
O(12 ⁱ)B—H(21 ⁱ)B—O(9 ⁱⁱ)A	179 (5)°
O(12 ⁱ)A—H(21 ⁱ)A—O(9 ⁱⁱⁱ)B	166 (5)

Symmetry code: (i) *x*, *y*, *z*; (ii) *x*, *y*, *z* - 2; (iii) *x* - ½, ½ - *y*, 2 - *z*.

duced by van der Waals interactions and the hydrogen bonds.

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Cristallochimie du Sélénium(+IV). III. Structure Cristalline de CuSe_2O_5

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CuSe_2O_5 , synthesized by solid-state reaction, crystallizes in the monoclinic system with $a=12.254$, $b=4.858$, $c=7.960$ Å, $\beta=110.7^\circ$; the space group is $C2/c$. The structure is characterized by zigzag strings of composition $(\text{CuSe}_2\text{O}_5)_n$, parallel to the c axis. In each string Cu is strongly bonded to four O atoms which form an approximately square-planar configuration. The Se atoms have a threefold coordination; two SeO_3 pyramids share a corner to form a $(\text{Se}_2\text{O}_5)^{2-}$ group. All selenium(+IV) lone pairs belong to a plane parallel to (100).

Dans le système CuO-SeO_2 , trois phases ont été mises en évidence à 450°C : CuSe_2O_5 , CuSeO_3 et Cu_2SeO_4 (Meunier, Bertaud & Galy, 1976). La détermination de la structure cristalline de CuSe_2O_5 fait suite à celle des phases VSe_2O_6 (Meunier, Bertaud & Galy, 1974) et ZnSe_2O_5 (Meunier & Bertaud, 1974). Elle entre ainsi dans le cadre général d'une étude sur la cristallochimie du sélénium(+IV) (Bertaud, 1974).

Rappel des données cristallographiques

CuSe_2O_5 cristallise dans le système monoclinique. Les groupes spatiaux possibles sont Cc ou $C2/c$. La densité mesurée par pycnométrie dans l'orthophtalate de diéthyle ($d=4,49 \pm 0,04$ g cm^{-3}) est en bon accord avec la densité calculée ($d_x=4,52$ g cm^{-3}) pour quatre motifs CuSe_2O_5 par maille (Tableau 1).

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Tableau 1. *Données radiocristallographiques de CuSe_2O_5*

Système monoclinique	Groupe spatial $C2/c$
a 12,254 (5) Å	d $4,49 \pm 0,04$ g cm^{-3}
b 4,858 (3)	d_x 4,52
c 7,960 (3)	Z 4
β 110,7 (1)°	

Détermination de la structure

La forme du cristal choisi pour la détermination structurale peut être assimilée à un prisme de section hexagonale, de hauteur 0,09 et de section 0,06 mm.

Les intensités des réflexions hkl ont été mesurées à l'aide d'un diffractomètre automatique CAD4 Enraf-Nonius (radiation $\text{Mo K}\alpha$). L'angle maximum de mesure était $\theta=27,5^\circ$. Les intensités des 462 réflexions indépendantes retenues pour l'affinement ont été corrigées par le facteur de Lorentz-polarisation. Les facteurs de diffusion des atomes de cuivre, de sélénium et d'oxygène ont été tirés des tables de Hanson, Herman, Lea & Skillman (1964). Les calculs ont été effectués sur Univac 1108.

La méthode d'addition symbolique utilisant le programme GAASA (Lindgren, Lindqvist & Nyborg, 1970) a été utilisée. Bien qu'il n'ait pas été possible de choisir entre les groupes Cc non-centrosymétrique et $C2/c$ centrosymétrique, à partir des tests statistiques effectués sur $|E|$, l'hypothèse du groupe $C2/c$ a été envisagée en premier.

Après traitement des données, il a été possible de déterminer la position des atomes lourds (Cu, Se). Après quelques cycles d'affinement, le facteur R était 0,17.