

The Crystal Structure of Manganese Nitrate Monohydrate

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Manganese nitrate monohydrate, $\text{Mn}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$, is orthorhombic, space group $C222_1$, with $a = 6.115$, $b = 13.244$, $c = 13.087$ Å, $Z = 8$. The structure was determined from X-ray diffractometer data by Patterson and Fourier syntheses, and refined by least-squares calculations to $R = 0.054$. The complex structure contains two crystallographically and chemically different coordination polyhedra. One Mn atom is six-coordinated by O atoms from four nitrate groups and from two water molecules, while the other is eight-coordinated by O atoms from four nitrate groups which are bonded bidentately. Both polyhedra have C_2 symmetry. The nitrate ligands link Mn atoms into a two-dimensional layer, held together in the third dimension by hydrogen bonds. The mean value of Mn–O bond lengths in the six-coordinated polyhedron is 2.196 Å, while the corresponding magnitude in the eight-coordinated polyhedron is 2.335 Å. From the geometry of the structure it can be concluded that the O atoms from nitrate groups are bonded to the metal ion by $\sigma(sp^2)$ bonds, and the O from the water molecule is bonded to the metal ion by a $\sigma(sp^3)$ bond.

Introduction

This crystal structure investigation was undertaken as part of a study of the structure of hydrated nitrates of bivalent metals. The existence of the monohydrated nitrate of manganese was established earlier (Weigel, Imelik & Prettre, 1964), as well as the monohydrated nitrates of some other bivalent metals, but the structures of these compounds have not yet been published. In order to obtain some information about the positions of water molecules in the coordination sphere for a nitrate complex with only one water molecule per formula unit, the structure of the title compound has been determined.

Experimental

Crystals of $\text{Mn}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ were prepared by heating the manganese nitrate tetrahydrate at 110°C. The crystals are pale-yellow cylinders, not longer than 1 mm. They are hygroscopic and had to be sealed in Lindemann capillary tubes for the crystal analysis. The initial unit-cell parameters were determined from oscillation and Weissenberg photographs; accurate unit-cell parameters were obtained later by a least-squares treatment of diffractometer data.

Crystal data

$\text{Mn}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$, $M_r = 197.0$. Orthorhombic, space group $C222_1$ (No. 20), $a = 6.115$ (2), $b = 13.244$ (6),

$c = 13.087$ (6) Å; $U = 1059.9$ (4) Å³, $Z = 8$, $D_x = 2.44$ g cm⁻³. $F(000) = 776$.

The intensity data were recorded by an ω -scan procedure on a Syntex $P\bar{1}$, four-circle computer-controlled diffractometer, with $\text{Mo } K\bar{\alpha}$ radiation from a graphite monochromator. The data were corrected for Lorentz and polarization effects, but not for absorption. The specimens were approximately spherical, size 0.2 mm. The final data set consisted of 727 independent reflexions with $|F_o| > 2\sigma(F_o)$.

Structure solution and refinement

At first, the structure was not solved in space group $C222_1$ but in space group $P2_1$ (No. 4), because, during the procedure for unit-cell determination by diffractometer, two possibilities for unit-cell selection were found: the first cell was orthorhombic with the periods given above, and the second cell was monoclinic with $a = 7.292$ (3), $b = 13.087$ (6), $c = 6.115$ (2) Å and $\beta = 114.73$ (3)°. We assumed that the orthorhombic symmetry was only apparent and that the crystal was really monoclinic, as was commonly found for the hydrated nitrates of bivalent metals solved previously. Therefore intensity data were collected for the monoclinic cell. The extinctions indicated two space groups: $P2_1$ or $P2_1/m$. The Patterson synthesis gave $P2_1$.

The structure was solved by standard Patterson and Fourier methods, and was refined by full-matrix least-squares calculations with the program *ORFLS* (Busing,

Martin & Levy, 1962). Refinement with anisotropic thermal parameters converged to $R = 0.054$. Scattering factors were taken for neutral-atom sites. The results of this calculation have been reported (Ribár, Milinski, Ćulum & Djurić, 1975; Milinski, 1976).

During the structure solution it was observed that the structure has two, mutually perpendicular axes of C_2 symmetry, which are not present in space group $P2_1$. Starting from this observation we again found the orthorhombic unit cell, as found earlier by experiment. The relations between the two cells are: $\mathbf{a} = \mathbf{c}_1$, $\mathbf{b} = 2\mathbf{a}_1 + \mathbf{c}_1$, $\mathbf{c} = \mathbf{b}_1$, where \mathbf{a}_1 , \mathbf{b}_1 , \mathbf{c}_1 are the vectors of elementary translations in the monoclinic representation of crystal space, and \mathbf{a} , \mathbf{b} , \mathbf{c} are the corresponding vectors in an orthorhombic representation. The indices of reflexions with respect to the monoclinic coordinate system H_1 , K_1 , L_1 were transformed into indices with respect to the orthorhombic system H , K , L by the rules: $H = L_1$, $K = 2H_1 + L_1$, $L = K_1$ (*International Tables for X-ray Crystallography*, 1952). In such a way, the set of 1243 measured reflexions was reduced to 727 independent reflexions. When two or more dependent reflexions were measured, their arithmetical mean value was used. The extinctions indicated the $C222_1$ space group.

While the asymmetric unit in space group $P2_1$ con-

tains two Mn atoms, four nitrate groups and two water molecules, all in general positions, the asymmetric unit in $C222_1$ contains two Mn atoms in special positions, and, in general positions, only two nitrate groups and one water molecule. After the transformation of atomic coordinates from $P2_1$ into $C222_1$, the least-squares refinement with anisotropic thermal parameters converged again to $R = 0.054$. The latter calculations were carried out on a Varian 73 computer at Novi Sad, with a block-diagonal least-squares procedure (Albano, Domenicano & Vaciago, 1966).

Final values of the atomic positional and thermal parameters (with respect to the unit cell in $C222_1$) are given in Table 1.*

Description of the structure and discussion

The structure consists of two crystallographically and chemically different coordination polyhedra. The first

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32298 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Fractional coordinates and anisotropic thermal parameters ($\times 10^4$)

Estimated standard deviations are given in parentheses. The anisotropic thermal parameters are given in the form:
 $T = \exp[-(b_{11}H^2 + b_{22}K^2 + b_{33}L^2 + b_{12}HK + b_{13}HL + b_{23}KL)]$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>b</i> ₁₁	<i>b</i> ₂₂	<i>b</i> ₃₃	<i>b</i> ₁₂	<i>b</i> ₁₃	<i>b</i> ₂₃
Mn(1)	1604 (3)	0000 (0)	5000 (0)	142 (4)	44 (1)	34 (1)	0 (0)	0 (0)	-3 (2)
Mn(2)	5000 (0)	-1424 (2)	2500 (0)	178 (4)	35 (1)	33 (1)	0 (0)	10 (4)	0 (0)
O(11)	-6177 (12)	1063 (5)	5937 (5)	210 (18)	48 (4)	28 (3)	-33 (15)	-13 (13)	-6 (6)
O(12)	-4423 (12)	2321 (6)	5254 (7)	250 (22)	45 (4)	76 (6)	-57 (15)	-89 (18)	2 (8)
O(13)	-6108 (14)	1217 (5)	4302 (5)	296 (25)	50 (4)	27 (3)	34 (17)	-46 (14)	-18 (6)
N(1)	-5482 (11)	1561 (6)	5149 (6)	132 (16)	39 (4)	36 (4)	0 (13)	-23 (14)	-1 (7)
O(21)	-1067 (13)	-0812 (6)	4015 (6)	240 (22)	54 (4)	51 (4)	16 (16)	-64 (17)	19 (8)
O(22)	-0570 (13)	0749 (6)	3710 (6)	288 (22)	53 (4)	43 (4)	-97 (16)	8 (17)	-12 (8)
O(23)	-2793 (11)	-0105 (6)	2768 (5)	213 (15)	61 (4)	34 (3)	-81 (17)	-54 (14)	3 (6)
N(2)	-1485 (10)	-0042 (6)	3505 (5)	123 (12)	44 (4)	27 (3)	-26 (18)	31 (12)	-9 (7)
OW	-2441 (15)	2426 (5)	2464 (8)	232 (16)	45 (3)	40 (3)	-17 (14)	6 (16)	0 (8)

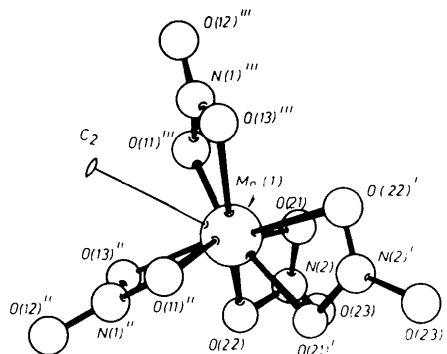


Fig. 1. Perspective view of the eight-coordination around Mn(1).

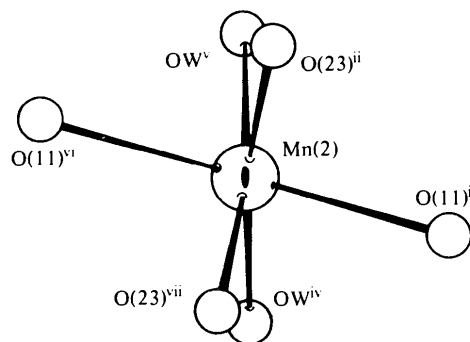


Fig. 2. Projection along the [010] axis of the six-coordinated polyhedron around Mn(2).

type of polyhedron is presented in Fig. 1. The central atom of this polyhedron is Mn(1). Both symmetrically independent nitrate groups are bonded to Mn(1) bidentately, and, by the symmetry axis C_2 which passes through Mn(1), the eightfold coordination of Mn is created. The geometry around Mn(1) can be described as a distorted dodecahedron, not particularly close to either of the common regular eightfold coordination polyhedra. The midpoints of the chelate ligands lie very close to the vertices of a tetrahedron, an effect which has been noticed in a few other eight-coordinated ions: $\text{Co}(\text{NO}_3)_4^{2-}$ (Bergman & Cotton, 1966) and $\text{Cd}(\text{NO}_3)_4^{2-}$ (Post & Trotter, 1974). For this and some spectroscopic reasons, these researchers regarded all these bidentate nitrate ligands as occupying only a single coordination site in the tetrahedron around the central atom.

Coordination of Mn by eight O atoms has been reported previously only in $(\text{Ph}_4\text{As})_2\text{Mn}(\text{NO}_3)_4$ (Drummond & Wood, 1970) with bonds in the range from 2.245 to 2.396 Å and with an atomic arrangement similar to that found here, possessing C_2 symmetry. Drummond & Wood do not propose that a bidentate ligand occupies a single coordination site, but that the above mentioned coordination is really dodecahedral.

The second type of coordination polyhedron is presented in Fig. 2, where atoms not bonded to the central Mn(2) are omitted. The four O atoms from the four nitrate groups, and two O atoms from water molecules form the vertices of a distorted octahedron around Mn(2). The octahedron possesses C_2 symmetry. The mean value of the bond lengths in the octahedron, 2.196 Å, is in good agreement with that commonly observed in six-coordinated Mn^{II} complexes, 2.1–2.2 Å. [An extensive review of the bibliography for six-coordinated Mn by O atoms is given by Popov, Herak, Prelesnik & Ribár (1973).]

All bonds around Mn(1) are longer than the bonds

around Mn(2), which is in agreement with a greater coordination number of the Mn(1) atom.

The nitrate groups bridge adjacent Mn atoms, as presented in Fig. 3, and their geometric distortions, which are not too great, can be interpreted as the influence of bonding to Mn.

An important property of the structure is that the two manganese atoms Mn(1) and Mn(2), bonded to the same nitrate group, lie close to the plane of that nitrate group, as shown in Table 2. On the other hand, the angles $\text{N}(1^{\text{ii}})\text{—O}(11^{\text{ii}})\text{—Mn}(2^{\text{i}}) = 121.2$ and $\text{N}(2)\text{—O}(23)\text{—Mn}(2^{\text{viii}}) = 123.8^\circ$ enable one to conclude that O(11ⁱⁱ) and O(23) form σ -bonds to Mn(2ⁱ) and Mn(2^{viii}) atoms, using a lone pair from sp^2 -hybrid orbitals of O (Cotton & Wilkinson, 1969a). The same conclusion seems plausible concerning the connexion of O atoms

Table 2. *Least-squares planes and atomic deviations* (Å)

X, Y, Z are in Å and related to the crystallographic axes.

Plane $P(1)$: N(1 ⁱⁱ), O(11 ⁱⁱ), O(12 ⁱⁱ), O(13 ⁱⁱ)			
$0.8310X - 0.5551Y - 0.0363Z = 0.8847$			
N(1 ⁱⁱ)	0.019	O(11 ⁱⁱ)	-0.006
O(12 ⁱⁱ)	-0.007	O(13 ⁱⁱ)	-0.006
Mn(1)	-0.307	Mn(2 ⁱ)	0.253
Plane $P(2)$: N(2), O(21), O(22), O(23)			
$0.7518X - 0.2298Y - 0.6181Z = -3.4945$			
N(2)	-0.011	O(21)	0.003
O(22)	0.004	O(23)	0.003
Mn(1)	0.187	Mn(2 ^{viii})	-0.393

Angles ($^\circ$) between planes and O—Mn bond directions

$P(1)\text{—O}(11^{\text{ii}})\text{—Mn}(1)$	-7.4	$P(1)\text{—O}(11^{\text{ii}})\text{—Mn}(2^{\text{i}})$	6.3
$P(1)\text{—O}(13^{\text{ii}})\text{—Mn}(1)$	-7.3	$P(2)\text{—O}(21)\text{—Mn}(1)$	4.3
$P(2)\text{—O}(23)\text{—Mn}(2^{\text{viii}})$	-10.0	$P(2)\text{—O}(22)\text{—Mn}(1)$	4.3

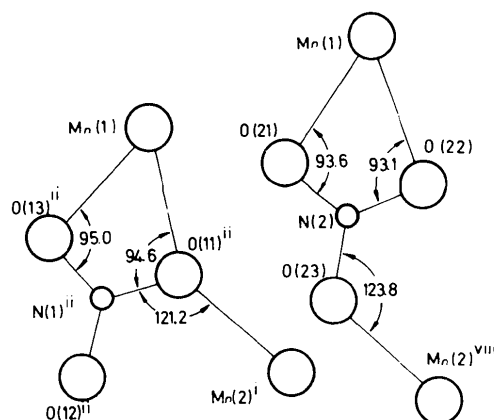


Fig. 3. Manner of bonding to Mn atoms for the two symmetrically independent nitrate groups.

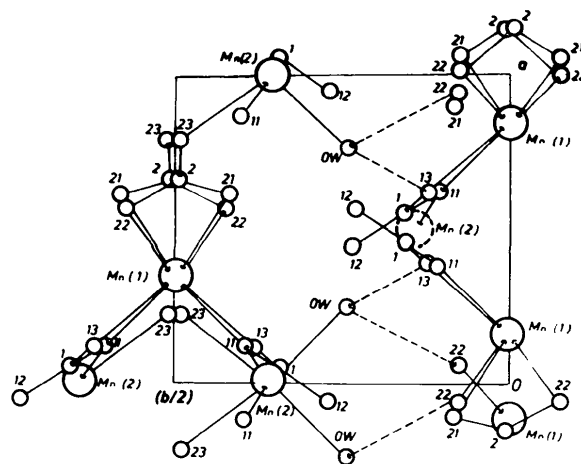


Fig. 4. Projection showing connexion between coordination polyhedra. Probable hydrogen bonds are marked by broken lines. The O and N labels and superscripts are omitted.

Table 3. Bond lengths (Å) and bond angles (°) with their e.s.d.'s in parentheses

The atoms N(1), O(11), O(12), O(13), assigned the same superscript, form one nitrate group around N(1), while the atoms N(2), O(21), O(22), O(23), assigned the same superscript, form a second nitrate group around N(2).

Symmetry code		
None	x, y, z	(v) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$
(i)	$x, -y, 1 - z$	(vi) $-x, -y, \frac{1}{2} - z$
(ii)	$1 + x, y, z$	(vii) $-x, y, \frac{1}{2} - z$
(iii)	$1 + x, -y, 1 - z$	(viii) $-x, y, z$
(iv)	$\frac{1}{2} + x, y - \frac{1}{2}, z$	(ix) $\frac{1}{2} - x, \frac{1}{2} + y, z$

Eight-coordinated polyhedron		Six-coordinated polyhedron	
Mn(1)—O(1 ⁱⁱ)	2.308 (9)	Mn(2)—O(1 ⁱⁱⁱ)	2.220 (9)
Mn(1)—O(13 ⁱⁱ)	2.322 (10)	Mn(2)—O(23 ⁱⁱ)	2.235 (8)
Mn(1)—O(21)	2.342 (10)	Mn(2)—O W^{iv}	2.133 (9)
Mn(1)—O(22)	2.367 (10)	Average	2.196 (9)
Average	2.335 (10)		
O(11 ⁱⁱ)—Mn(1)—O(13 ⁱⁱ)	55.3 (4)	O(11 ⁱⁱⁱ)—Mn(2)—O(23 ^{vii})	77.3 (3)
O(11 ⁱⁱ)—Mn(1)—O(21)	169.2 (3)	O(11 ⁱⁱⁱ)—Mn(2)—O(23 ⁱⁱ)	83.3 (3)
O(11 ⁱⁱ)—Mn(1)—O(22)	117.0 (4)	O(11 ⁱⁱⁱ)—Mn(2)—O(11 ^{vi})	155.1 (3)
O(11 ⁱⁱ)—Mn(1)—O(21 ⁱ)	80.6 (4)	O(11 ⁱⁱⁱ)—Mn(2)—O W^{iv}	111.1 (4)
O(11 ⁱⁱ)—Mn(1)—O(22 ⁱ)	101.9 (3)	O(11 ⁱⁱⁱ)—Mn(2)—O W^{iv}	87.0 (4)
O(11 ⁱⁱ)—Mn(1)—O(11 ⁱⁱⁱ)	108.0 (3)	O(23 ⁱⁱ)—Mn(2)—O(23 ^{vii})	77.2 (3)
O(11 ⁱⁱ)—Mn(1)—O(13 ⁱⁱⁱ)	82.0 (3)	O(23 ⁱⁱ)—Mn(2)—O W^{iv}	169.8 (4)
O(13 ⁱⁱ)—Mn(1)—O(21)	121.5 (3)	O(23 ⁱⁱ)—Mn(2)—O W^{iv}	97.6 (4)
O(13 ⁱⁱ)—Mn(1)—O(22)	76.5 (4)	O W^{iv} —Mn(2)—O W^{iv}	88.9 (4)
O(13 ⁱⁱ)—Mn(1)—O(21 ⁱ)	108.5 (4)		
O(13 ⁱⁱ)—Mn(1)—O(22 ⁱ)	155.5 (3)	Hydrogen bonds	
O(13 ⁱⁱ)—Mn(1)—O(13 ⁱⁱⁱ)	105.9 (3)	O W^{ii} —O(13 ^{vii})	2.948 (15)
O(21)—Mn(1)—O(22)	53.7 (3)	O W^{ii} —O(22 ⁱⁱ)	2.983 (15)
O(21)—Mn(1)—O(22 ⁱ)	79.0 (3)	O(13 ^{vii})—O W^{ii} —O(22 ⁱⁱ)	98.0 (4)
O(21)—Mn(1)—O(21 ⁱ)	91.6 (4)	O(13 ^{vii})—O W^{ii} —Mn(2 ^{ix})	128.0 (4)
O(22)—Mn(1)—O(22 ⁱ)	111.6 (3)	O(22 ⁱⁱ)—O W^{ii} —Mn(2 ^{ix})	104.5 (5)
		Sum of angles	330.5 (15)
First (NO ₃) group		Second (NO ₃) group	
N(1)—O(11)	1.296 (13)	N(2)—O(21)	1.245 (14)
N(1)—O(12)	1.205 (13)	N(2)—O(22)	1.218 (14)
N(1)—O(13)	1.258 (14)	N(2)—O(23)	1.256 (14)
Average	1.253 (13)	Average	1.240 (14)
O(11)—N(1)—O(12)	120.7 (10)	O(21)—N(2)—O(22)	119.5 (10)
O(11)—N(1)—O(13)	114.6 (10)	O(21)—N(2)—O(23)	119.2 (10)
O(12)—N(1)—O(13)	124.5 (10)	O(22)—N(2)—O(23)	121.3 (10)
Sum of angles	359.8 (30)	Sum of angles	360.0 (30)
Angles of bonds from oxygen to nitrogen and metal			
N(1 ⁱⁱ)—O(11 ⁱⁱ)—Mn(2 ⁱ)	121.2 (5)	N(1 ⁱⁱⁱ)—O(11 ⁱⁱⁱ)—Mn(1)	94.6 (4)
N(2)—O(23)—Mn(2)	123.8 (5)	N(1 ⁱⁱⁱ)—O(13 ⁱⁱⁱ)—Mn(1)	95.0 (4)
N(2)—O(21)—Mn(1)	93.6 (4)	N(2)—O(22)—Mn(1)	93.1 (4)

to Mn(1), although the corresponding angles N(1ⁱⁱ)—O(11ⁱⁱ)—Mn(1) *etc.* are not very close to 120°, as would be necessary (Cotton & Wilkinson, 1969*b*), but it is evident that the bidentate bonding causes this difference.

The arrangement of polyhedra in the unit cell is presented in Fig. 4. The centres of eight-coordinated polyhedra, Mn(1) atoms, are placed on C_2 symmetry axes parallel to the x axis, and the centres of six-coordinated polyhedra, Mn(2) atoms, are placed on C_2 axes parallel to the y axis of the unit cell. The bridging nitrate groups

link polyhedra in layers parallel to the (010) crystallographic plane. The layers are held together in the third dimension by hydrogen bonds. Although the difference Fourier map did not give precise locations of H atoms, from the geometry of the structure it clearly follows that the hydrogen bonds radiate from atom OW to O(13) and O(22), as shown in Fig. 4, with the bond lengths and bond angles given in Table 3.

The hydrogen bonds described satisfy the conditions for belonging to class 1' type J , in the

classification of water molecules in crystalline hydrates (Ferraris & Franchini-Angela, 1972). In this class, O forms a donor coordination bond to a bivalent metallic ion, using a lone pair from the sp^3 -hybrid electron orbitals.

The bond between Mn(2) and OW, 2.133 Å, is significantly shorter than the bond between Mn(2) and O(11) and O(23) from the nitrate groups, 2.242 and 2.234 Å. So we can conclude that the donor coordination bond of O to a metallic ion is shorter in the case when this bond is formed by a lone pair from an sp^3 -orbital, than in the case when the bond is formed by a lone pair from an sp^2 -orbital.

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Structure Cristalline et Moléculaire du Diméthoxy-3 β e,16 β e Oléanène-12(13) Olide-28-21 β a

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The crystal structure of the triterpene $C_{32}H_{50}O_4$ extracted from the bark of roots of several *Albizzia* has been determined. From three-dimensional data collected with Cu $K\alpha$ radiation, the space group has been found to be $P2_12_12_1$, [$Z = 4$, $a = 9.034$ (5), $b = 25.668$ (7), $c = 12.161$ (8) Å]. The structure has been solved by direct methods and refined to $R = 0.067$ for 2464 observed reflexions. The results confirm the structure to be that previously assumed by chemical investigations, i.e. 3 β e,16 β e-dimethoxyolean-12-en-28-21 β a-olide. The calculation of dihedral angles between the H atoms at 21 and 22a-22e supports the results previously obtained by NMR spectroscopy for the triterpenic lactone.

Introduction

L'étude chimique de sept mimosacées de l'Afrique de l'Ouest (*Albizzia adianthifolia*, *A. Ferruginéa*, *A. Zigya*,

A. Glabérima, *A. Boromensis*, *A. stipulata* et *Samanéa dinklagei*), utilisées pour la plupart comme médicaments, a permis à l'un d'entre nous (Comeau, 1972) d'isoler dans chacune de ces espèces un même