

The Crystal Structure of 4-Acetyl-2'-nitrobiphenyl

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4-Acetyl-2'-nitrobiphenyl, $C_{14}H_{11}O_3N$, is monoclinic, space group $P2_1/c$, with $a=12.146$ (1), $b=12.647$ (1), $c=7.812$ (1) Å, $\beta=94.33$ (1)°, $Z=4$, $D_m=1.29$, $D_c=1.34$ g cm⁻³. The structure was solved by direct methods and refined by block-diagonal and full-matrix least-squares calculations to $R=4.5\%$ for 1783 structure factors. The average ring C-C bond is 1.386 Å. The molecule is non-planar; there are rotations about the phenyl bond and C(10)-C(13) bond of +45.9 (2) and +12.7 (2)° respectively with additional small rotations. There is also distortion in the bond angles around C(2) and N.

Experimental

Cell dimensions were obtained by least-squares refinement of 20 high-angle reflexions measured on a diffractometer with Cu $K\alpha$ radiation. The observed density was measured by flotation in aqueous cadmium *n*-dodecatungstaborate.

Crystal data

$C_{14}H_{11}O_3N$, $M=241.2$. Monoclinic,
 $a=12.146$ (1), $b=12.647$ (1), $c=7.812$ (1) Å,
 $\beta=94.33$ (1)°, $U=1196.6$ Å³, $D_m=1.29$ g cm⁻³,
 $D_c=1.34$ g cm⁻³, $Z=4$, $F(000)=504$, $\lambda(\text{Cu } K\alpha)=1.5418$ Å.

Absent spectra: $0k0$ with k odd and $h0l$ with l odd. Space group $P2_1/c$.

A suitable single crystal was mounted about the needle axis, corresponding to the c axis of the unit cell. Intensities of 1783 reflexions with $\theta < 60^\circ$ of which 1622 were observed were collected on a Siemens single-crystal diffractometer AED with Cu $K\alpha$ radiation (Skapski & Troughton, 1970).

The data were corrected for Lorentz and polarization effects; overall scale and temperature factors were obtained by Wilson's (1942) method, and a list of normalized structure factors, calculated without ϵ , produced.

The structure was solved by direct methods from the 1121 relationships with probability $> 90\%$ obtained for the 140 reflexions with $E > 1.75$. An E map based on the 136 determined phases showed the positions of all the non-hydrogen atoms. After block-diagonal least-squares refinement to $R=0.095$ where $R=\frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$, all hydrogen atoms were located from a difference synthesis. Full-matrix least-squares refinement of positional, scale and anisotropic thermal parameters for C, N and O and isotropic thermal

parameters for H resulted in a final $R=0.045$. * Atomic scattering factors were obtained from Doyle & Turner (1968) except those for hydrogen which came from Stewart, Davidson & Simpson (1965).

Table 1 gives the heavy-atom coordinates and Table 2 their thermal parameters. The hydrogen-atom parameters are quoted in Table 3. The numbering of the atoms in the molecule and a stereo view of the structure are shown in Figs. 1 and 2 (Johnson, 1965).

All intermolecular contacts of less than 3.5 Å are shown in Table 4; the bond lengths and angles with

Table 1. Heavy-atom coordinates

Fractional coordinates ($\times 10^4$) of the heavy atoms with their estimated standard deviations in parentheses.

| | X/a | Y/b | Z/c |
|-------|----------|----------|----------|
| O(1) | -506 (2) | 5073 (2) | 2813 (3) |
| O(2) | 772 (2) | 4451 (2) | 1330 (3) |
| O(3) | 4909 (2) | 1074 (2) | 3410 (4) |
| N | 447 (2) | 5045 (2) | 2413 (3) |
| C(1) | 2285 (2) | 5456 (2) | 3858 (3) |
| C(2) | 1234 (2) | 5796 (2) | 3276 (4) |
| C(3) | 887 (3) | 6831 (3) | 3397 (5) |
| C(4) | 1612 (4) | 7578 (3) | 4100 (5) |
| C(5) | 2662 (4) | 7286 (3) | 4690 (5) |
| C(6) | 2994 (3) | 6241 (2) | 4578 (4) |
| C(7) | 2677 (2) | 4344 (2) | 3829 (3) |
| C(8) | 3722 (2) | 4133 (2) | 3279 (4) |
| C(9) | 4110 (2) | 3111 (2) | 3246 (4) |
| C(10) | 3474 (2) | 2270 (2) | 3743 (3) |
| C(11) | 2437 (2) | 2481 (2) | 4315 (4) |
| C(12) | 2048 (2) | 3503 (2) | 4362 (4) |
| C(13) | 3936 (2) | 1179 (2) | 3642 (4) |
| C(14) | 3195 (3) | 244 (3) | 3808 (5) |

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30382 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. Thermal parameters ($\times 10^3 \text{ \AA}^2$) with *e.s.d.*'s in parentheses

Thermal parameters are defined as
 $\exp [-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

| | U_{11} | U_{22} | U_{33} | $2U_{12}$ | $2U_{13}$ | $2U_{23}$ |
|-------|----------|----------|----------|-----------|-----------|-----------|
| O(1) | 44 (1) | 123 (2) | 105 (2) | 8 (3) | 27 (2) | -28 (3) |
| O(2) | 59 (1) | 82 (2) | 73 (1) | 28 (2) | -8 (2) | -34 (3) |
| O(3) | 64 (2) | 72 (2) | 162 (3) | 49 (2) | 70 (3) | 3 (3) |
| N | 47 (1) | 68 (2) | 62 (2) | 26 (2) | 2 (2) | 13 (3) |
| C(1) | 51 (2) | 47 (2) | 52 (2) | 2 (3) | 12 (3) | 10 (3) |
| C(2) | 52 (2) | 51 (2) | 54 (2) | 13 (3) | 18 (3) | 8 (3) |
| C(3) | 69 (2) | 66 (2) | 73 (2) | 42 (4) | 30 (4) | 22 (4) |
| C(4) | 109 (3) | 45 (2) | 86 (3) | 17 (4) | 40 (5) | 4 (4) |
| C(5) | 101 (3) | 53 (2) | 82 (3) | -9 (4) | 11 (4) | -3 (4) |
| C(6) | 74 (2) | 50 (2) | 71 (2) | -12 (3) | -8 (3) | 5 (3) |
| C(7) | 42 (1) | 46 (2) | 47 (2) | 0 (2) | -5 (2) | 3 (2) |
| C(8) | 38 (1) | 53 (2) | 63 (2) | -8 (3) | 5 (3) | 14 (3) |
| C(9) | 35 (1) | 61 (2) | 61 (2) | 8 (3) | 6 (3) | 4 (3) |
| C(10) | 42 (1) | 50 (2) | 47 (1) | 13 (2) | -3 (2) | 3 (2) |
| C(11) | 46 (2) | 47 (2) | 54 (2) | -4 (3) | 15 (3) | 9 (3) |
| C(12) | 42 (2) | 52 (2) | 56 (2) | 9 (3) | 22 (3) | 5 (3) |
| C(13) | 56 (2) | 57 (2) | 62 (2) | 25 (3) | 6 (3) | -3 (3) |
| C(14) | 71 (2) | 45 (2) | 82 (3) | 15 (3) | -6 (4) | -12 (3) |

some of the intramolecular non-bonded distances are in Table 5. The equations of the ring systems C(1)–C(6) and C(7)–C(12), and the groups C(2), N, O(1), O(2), and C(10), C(13), C(14), O(3), calculated with respect to the orthogonal triad a', b', c' , where b' coincides with b , a' is the projection of a on a plane perpendicular to b' , and c' is perpendicular to a' and b' , with the deviations of the atoms from the plane, are given in Table 6.

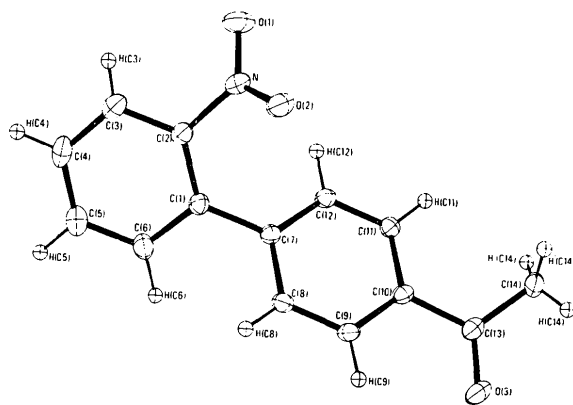


Fig. 1. The molecular structure showing thermal vibration ellipsoids scaled to enclose 50% probability. The hydrogen atoms were assigned B 's of 2.0 \AA^2 for illustrative purposes.

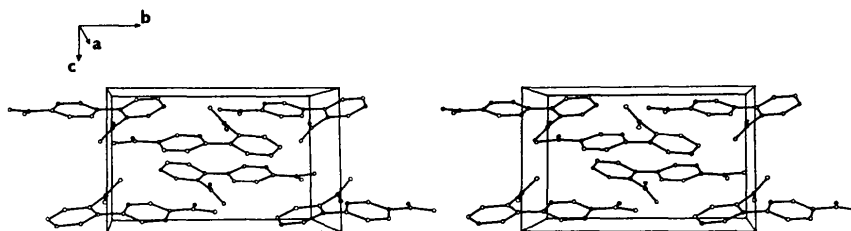


Fig. 2. A stereoscopic view of the molecular packing.

Table 3. Fractional coordinates and thermal parameter (all $\times 10^3$) of the hydrogen atoms

| | X/a | Y/b | Z/c | U_{1so} |
|----------|-------|-------|-------|-----------|
| H(C3) | 22 | 697 | 306 | 62 |
| H(C4) | 136 | 827 | 415 | 84 |
| H(C5) | 320 | 782 | 555 | 117 |
| H(C6) | 376 | 600 | 525 | 120 |
| H(C8) | 415 | 469 | 281 | 62 |
| H(C9) | 480 | 296 | 275 | 64 |
| H(C11) | 197 | 192 | 468 | 65 |
| H(C12) | 131 | 366 | 481 | 50 |
| H(C14) | 355 | -27 | 359 | 98 |
| H'(C14) | 263 | 25 | 277 | 148 |
| H''(C14) | 285 | 22 | 499 | 117 |

Table 4. Intermolecular distances between atoms less than 3.5 \AA apart (excluding hydrogen atoms)

| i | j | Equipoint indication | d_{ij} (\AA) |
|-------|-------|---------------------------------------|---------------------------|
| O(2) | O(2) | $-x \ 1-y \ -z$ | 3.028 |
| N | O(2) | $-x \ 1-y \ -z$ | 3.239 |
| O(2) | O(1) | $-x \ 1-y \ -z$ | 3.284 |
| C(8) | O(3) | $1-x \ \frac{1}{2}+y \ \frac{1}{2}-z$ | 3.296 |
| O(1) | C(14) | $-x \ \frac{1}{2}+y \ \frac{1}{2}-z$ | 3.419 |
| C(2) | O(1) | $-x \ 1-y \ 1-z$ | 3.425 |
| C(14) | O(3) | $1-x \ -y \ 1-z$ | 3.471 |
| C(12) | O(1) | $-x \ 1-y \ 1-z$ | 3.500 |

Discussion

The average C–C bond in the two phenyl rings is 1.386 \AA with no bond differing from this by more than 3σ . The angles C(1)–C(2)–C(3) and C(3)–C(2)–N, 123.3 and 116.8° respectively, are similar to the values 123.7 and 117.1° in *o*-nitrobenzoic acid (Tavale & Pant, 1973).

The C(2)–N bond, $1.475(4) \text{ \AA}$, is not significantly different from the values $1.478(13) \text{ \AA}$ in *o*-nitrobenzoic acid, $1.470(12)$ and $1.482(12) \text{ \AA}$ in 1-(2',4'-dinitrophenyl)-4-chloropyrazole (Galigné & Falgouttes, 1970). The N–O bonds $1.222(3)$ and $1.219(3) \text{ \AA}$ are comparable with the values quoted for similar nitro-substituted compounds; tri-*p*-nitrophenyl phosphate (Mazhur-ul-Haque & Caughlan, 1970), $1.22(1) \text{ \AA}$; *o*-nitrobenzoic acid, $1.198(13)$ and $1.221(13) \text{ \AA}$. The angles C(2)–N–O(1), C(2)–N–O(2) and O(1)–N–O(2), $117.3(3)$, $118.8(2)$ and $123.9(3)^\circ$ are not significantly different from the values in *o*-nitrobenzoic acid and 1-(2',4'-dinitrophenyl)-4-chloropyrazole.

The distribution of bond angles around C(1) is similar to that for other 2'-halogen-substituted biphenyls with the angle C(2)–C(1)–C(6) considerably less than 120°

and C(7)–C(1)–C(2) greater than 120° . In the present structure the angle C(2)–C(1)–C(7) $125.2(3)^\circ$ does not differ significantly from $125.4(15)^\circ$ in 2'-iodobiphenyl-4-carboxylic acid (Sutherland, 1970) and $124.6(9)^\circ$ in *o*-nitrobenzoic acid; the angle C(2)–C(1)–C(6) of

$115.6(3)^\circ$ is not significantly different from $117.9(16)^\circ$ and $118.7(9)^\circ$ in 2'-iodobiphenyl-4-carboxylic acid and *o*-nitrobenzoic acid respectively. In several of the 2'-halogen-substituted biphenyls the halogen atom has been found to be displaced out of the plane C(1)–C(6); the nitrogen atom in the present structure is displaced by -0.083 \AA , the C(2)–N bond being inclined at 3.2° to the biphenyl ring; C(7) is displaced from the ring C(1)–C(6) by 0.053 \AA .

Table 5. Bond lengths (\AA) and bond angles ($^\circ$), *e.s.d.*'s in parentheses

| | | | |
|----------------|-----------|-------------------|-----------|
| O(1)—O(2) | 2.154 (3) | O(2)—N | 1.219 (3) |
| O(1)—N | 1.222 (3) | O(2)—C(2) | 2.322 (4) |
| O(1)—C(2) | 2.307 (3) | O(2)—C(1) | 2.889 (3) |
| O(1)—C(3) | 2.811 (4) | O(2)—C(7) | 2.916 (3) |
| C(1)—C(2) | 1.390 (4) | O(2)—C(12) | 2.983 (3) |
| C(1)—C(6) | 1.404 (4) | N—C(2) | 1.475 (4) |
| C(1)—C(7) | 1.485 (4) | N—C(3) | 2.433 (4) |
| C(2)—C(3) | 1.381 (4) | N—C(1) | 2.480 (3) |
| C(3)—C(4) | 1.376 (5) | N—C(7) | 2.982 (3) |
| C(4)—C(5) | 1.374 (6) | N—C(12) | 3.075 (4) |
| C(5)—C(6) | 1.386 (5) | C(13)—C(14) | 1.498 (5) |
| C(7)—C(8) | 1.396 (4) | C(13)—O(3) | 1.216 (4) |
| C(7)—C(12) | 1.392 (4) | C(14)—O(3) | 2.373 (5) |
| C(8)—C(9) | 1.376 (4) | C(14)—C(10) | 2.585 (4) |
| C(9)—C(10) | 1.387 (4) | C(14)—C(11) | 3.010 (5) |
| C(10)—C(11) | 1.394 (4) | O(3)—C(10) | 2.337 (3) |
| C(10)—C(13) | 1.494 (4) | O(3)—C(9) | 2.753 (4) |
| C(11)—C(12) | 1.378 (4) | | |
| O(1)—N—O(2) | 123.9 (3) | C(1)—C(7)—C(8) | 119.1 (3) |
| O(1)—N—C(2) | 117.3 (3) | C(1)—C(7)—C(12) | 122.3 (3) |
| O(2)—N—C(2) | 118.8 (2) | C(8)—C(7)—C(12) | 118.5 (3) |
| N—C(2)—C(3) | 116.8 (3) | C(7)—C(8)—C(9) | 120.4 (3) |
| N—C(2)—C(1) | 119.8 (3) | C(8)—C(9)—C(10) | 121.1 (3) |
| C(1)—C(2)—C(3) | 123.3 (3) | C(9)—C(10)—C(11) | 118.6 (3) |
| C(2)—C(3)—C(4) | 119.2 (3) | C(9)—C(10)—C(13) | 118.3 (3) |
| C(3)—C(4)—C(5) | 120.0 (3) | C(11)—C(10)—C(13) | 123.1 (3) |
| C(4)—C(5)—C(6) | 120.2 (3) | C(10)—C(11)—C(12) | 120.5 (3) |
| C(5)—C(6)—C(1) | 121.8 (3) | C(11)—C(12)—C(7) | 120.8 (3) |
| C(6)—C(1)—C(2) | 115.6 (3) | C(10)—C(13)—C(14) | 119.6 (3) |
| C(6)—C(1)—C(7) | 119.2 (3) | C(10)—C(13)—O(3) | 118.9 (3) |
| C(2)—C(1)—C(7) | 125.2 (3) | O(3)—C(13)—C(14) | 121.6 (3) |

Table 6. The mean planes of the systems

(I) C(1)–C(6); (II) C(7)–C(12); (III) C(2), N, O(1), O(2);
(IV) C(10), C(13), C(14), O(3)

Equations of the planes

| | |
|-------|--|
| (I) | $-0.3896X' - 0.1847Y' + 0.9023Z' = 0.4449$ |
| (II) | $0.3361X' + 0.1008Y' + 0.9364Z' = 4.3713$ |
| (III) | $0.1754X' - 0.6804Y' + 0.7116Z' = -2.9402$ |
| (IV) | $0.1457X' + 0.0000Y' + 0.9893Z' = 3.4682$ |

Deviations from the mean planes in $\text{\AA} (\times 10^3)$

| | I | II | III | IV |
|-------|------|-------|-------|-------|
| O(1) | 652 | -1935 | -2 | -1414 |
| O(2) | -884 | -2545 | -2 | -2318 |
| O(3) | -544 | 189 | 4917 | -1 |
| N | -83 | -1834 | 7 | -1550 |
| C(1) | 0 | -5 | 831 | -123 |
| C(2) | -5 | -804 | -2 | -753 |
| C(3) | 6 | -728 | -901 | -722 |
| C(4) | -2 | 162 | -1007 | -58 |
| C(5) | -3 | 972 | -211 | 577 |
| C(6) | 4 | 895 | 699 | 551 |
| C(7) | 53 | -8 | 1855 | -77 |
| C(8) | -791 | 2 | 1961 | -311 |
| C(9) | -760 | 6 | 2904 | -267 |
| C(10) | 98 | -8 | 3763 | -1 |
| C(11) | 955 | 2 | 3672 | 251 |
| C(12) | 934 | 6 | 2735 | 218 |
| C(13) | 60 | -30 | 4745 | 4 |
| C(14) | 750 | -344 | 5482 | -1 |

In common with the halogen-substituted biphenyls not only is there a rotation of one ring with respect to the other by an angle φ_1 about C(1)–C(7) but also two other rotations, φ_2 of the ring C(1)–C(6) about an axis which is in the plane of that ring and passes through C(1) perpendicular to C(1)–C(7) and φ_3 of the ring C(7)–C(12) about an axis which is in the plane of that ring and passes through C(7) perpendicular to C(1)–C(7). In the present structure the angles φ_1 , φ_2 and φ_3 are respectively $+45.9(2)$, $+2.1(2)$ and $-0.2(1)^\circ$.

The C(1)–C(7) length $1.485(4) \text{ \AA}$ and the C(10)–C(13) length $1.494(4) \text{ \AA}$ do not differ significantly from the theoretical value of 1.477 \AA quoted by Cruickshank & Sparks (1960) for the length between trigonally hybridized carbon atoms.

The C(13)–O(3) and C(13)–C(14) bonds $1.216(4)$ and $1.498(5) \text{ \AA}$ respectively compare favourably with 1.23 and 1.50 \AA , the quoted values for C–O and C–CH₃ bonds, and do not differ significantly from the respective values of $1.211(9)$ and $1.495(13) \text{ \AA}$ obtained for 4-acetyl-2'-fluorobiphenyl (Young, Tollin & Sutherland, 1968).

The acetyl group is rotated about the C(10)–C(13) bond through an angle φ_4 of $+12.7(2)^\circ$. Associated with this is a second rotation $\varphi_5 = +1.2(2)^\circ$ of the group C(10), C(13), C(14) and O(3) about an axis which is in the plane of that group and passes through C(10) perpendicular to C(7)–C(10). The angle φ_4 is significantly larger than that obtained in 2'-halogen-substituted biphenyls.

In the solid phase biphenyl is planar; the introduction of the nitro group in the 2' position results in intramolecular overcrowding. The strain is partly relieved by a twisting of the rings about the phenyl bond, φ_1 with the additional small rotations φ_2 and φ_3 . There is in addition an increase in the C(7)–C(1)–C(2) bond angle; the nitrogen atom is displaced out of the plane of the phenyl ring away from the displacement of C(7); the nitro group is rotated about the C(2)–N bond away from the phenyl plane, with the N–H(12) distance 2.716 \AA comparing well with the value of 2.70 \AA obtained from the van der Waals radii. It is probable that there is a relayed steric effect which results in a small rotation and displacement of the acetyl group. The large value of φ_4 is due partly to this and partly to intermolecular forces.

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Etudes Structurales de Composés Oxyfluorés du P^V. II. Structure Cristalline de Na₂PO₃F β

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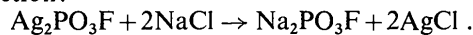
The crystal structure of Na₂PO₃F β has been studied by X-ray diffraction. This compound is orthorhombic, space group *P*2₁2₁. The cell dimensions are $a = 5.416 \pm 0.003$, $b = 6.929 \pm 0.004$, $c = 19.079 \pm 0.010$ Å and $Z = 8$. Data were measured on an automatic diffractometer using Cu *K*α radiation, $\lambda = 1.5418$ Å. The structure was established by statistical methods and refined by least-squares analysis using anisotropic thermal parameters. The structure is characterized by two kinds of PO₃F²⁻ tetrahedron. The environments of oxygen and fluorine atoms are described. The final *R* index is 0.052 for 712 observed reflexions.

Introduction

Dans le cadre d'une étude systématique de la stéréochimie de l'ion PO₃F²⁻ nous avons déjà publié la structure de LiKPO₃F·H₂O (Galigné, Durand & Cot, 1974). La présente étude concerne la structure détaillée de Na₂PO₃F β dans laquelle n'intervient aucune liaison hydrogène.

Partie expérimentale

Le monofluorophosphate de sodium est préparé suivant la réaction:



Par cristallisation entre 20 et 40°C d'une solution aqueuse il se forme des monocristaux de monofluorophosphate de sodium anhydre que nous appelons Na₂PO₃F β (Durand, Granier & Cot, 1973).

Une étude au rétigraphe de Rimsky nous a permis de préciser le système cristallographique, le groupe d'espace et les paramètres de la maille. Ces derniers

sont affinés à partir des diagrammes de poudre. Les résultats sont les suivants:

$$\begin{aligned} a &= 5,416 \pm 0,003 \text{ \AA} & \rho_x &= 2,676 \pm 0,005 \text{ g cm}^{-3} \\ b &= 6,929 \pm 0,004 & \rho_{\text{obs}} &= 2,674 \pm 0,006 \text{ g cm}^{-3} \\ c &= 19,079 \pm 0,010 \\ V &= 716,1 \pm 1,3 \text{ \AA}^3 & Z &= 8 \end{aligned}$$

Système orthorhombique, groupe d'espace *P*2₁2₁. Axe d'allongement: *a*. La masse volumique ρ_{obs} est mesurée par pycnométrie dans le benzène à 20,0 ± 0,1°C

Un monocristal a été sélectionné puis transformé en sphère ($R = 0,35\text{mm}$). Les mesures d'intensité ont été effectuées à la température ambiante sur diffractomètre automatique Enraf-Nonius type CAD 3. Un balayage $\theta/2\theta$ est utilisé avec le rayonnement Cu *K*α monochromatisé. 712 réflexions indépendantes ont été mesurées jusqu'à un angle $\theta = 68^\circ$. La réflexion de référence 2 $\bar{1}$ 2 était repérée à intervalles réguliers de 50 réflexions et n'a pas montré de variations supérieures à 3%. Les intensités ont été corrigées des effets