(4) (Marchand, Rajapaksa, Reddy, Watson & Nagl, 1989) have been reported. The synthesis and chemistry of compound (2) and related materials have been reported (Marchand, Arney, Gilardi & Flippen-Anderson, 1987; Marchand, Annapurna, Reddy, Watson & Nagl, 1989).

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N-(Diméthyl-4,6 pyridyl-2) (Nitro-4 phényl)-2 Propionamide

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Abstract. $C_{16}H_{17}N_3O_3$, $M_r = 299{\cdot}3$, monoclinic, $P2_1/n$, $a = 10{\cdot}015\ (3)$, $b = 11{\cdot}531\ (3)$, $c = 13{\cdot}681\ (2)$ Å, $\beta = 107{\cdot}14\ (2)°$, $V = 1510\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}317$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}087$ mm^{-1}, $F(000) = 632$, $T = 294\ (1)$ K, $R = 0{\cdot}045$ for 930 independent reflections. There is a delocalized orbital along the amide group. The least-squares planes of the two rings make an angle of 81·2 (2)°. The atoms of the nitro group are very near to the phenyl-ring plane and those of the amide group are near to the pyridyl ring plane. The molecules form layers which spread out along the $z = 0$ and $z = \frac{1}{2}$ planes. Two molecules in two neighbouring layers are linked by an N—H···O hydrogen bond. The title compound belongs to a group of drugs with anti-inflammatory properties. It has been synthetized and studied in order to adjust the pharmacological activity of *N*-(4,6-dimethyl-2-pyridyl)benzamide, which is the typical compound of the group.

Partie expérimentale. Cristal approximativement parallélépipédique: 0,08 × 0,15 × 0,35 mm. Dimensions de la maille déterminées avec 25 réflexions telles que $5{,}24 \le \theta \le 17{,}90°$. Diffractomètre Enraf–Nonius CAD-4. $0{,}049 \le (\sin\theta)/\lambda \le 0{,}527$ Å^{-1}, $0 \le h \le 13$, $0 \le k \le 12$ et $-14 \le l \le 13$. Réflexions de contrôle de l'intensité: 211, $\bar{3}1\bar{4}$ et $22\bar{2}$. Pas de variation significative des intensités au cours des mesures. $\sigma(I)/I$ (contrôle) = 0,0018. 1853 réflexions indépendantes. 923 réflexions inobservées [$I < 1{,}5\sigma(I)$]. Pas de correction d'absorption. Méthodes directes, programme *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Affinement sur F, programme à matrice complète. Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974). H liés à N(7), C(16), C(17) et C(19): série de Fourier des ΔF. Autres H: positions calculées. Paramètres affinés: x, y, z et β_{ij} de C, N et O. Paramètres de position et d'agitation des H non affinés (nombre insuffisant de réflexions observées). $B(\mathrm{H}) = B_{\text{éq}}$ de l'atome lié à H + 1 Å^2. $R = 0{,}045$, $wR = 0{,}041$, $w = 1/\sigma^2(F)$, $S = 1{,}13$, $(\Delta/\sigma)_{\max} < 0{,}01$, $|\Delta\rho|_{\max} = 0{,}17\ (4)$ e Å^{-3}. Coefficient d'extinction secondaire isotrope $g = 3{,}5\ (8) \times 10^{-7}$. Programmes de calcul du système *SDP* (B. A. Frenz & Associates Inc., 1982). Angles des plans moyens: programme

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BP7C (Ito & Sugawara, 1983). Angles de torsion: programme *ORFFE* (Busing, Martin & Levy, 1964). Fig. 1 et 2: programme *ORTEP*II (Johnson, 1976).

Les coordonnées atomiques relatives et les facteurs de température isotropes équivalents sont rapportés dans le Tableau 1,* les longueurs et les angles des liaisons dans le Tableau 2. La Fig. 1 représente la molécule vue en perspective et la Fig. 2 la structure vue selon [010].

Littérature associée. Structure de l'acide (isobutyl-4 phényl)-2 propionique (McConnell, 1974). Ce composé, appelé aussi ibuprofen ou prufen, est un antiinflammatoire. Structure cristalline du *N*-(diméthyl-4,6 pyridinyl-2) benzamide (Rodier, Piessard, Le Baut & Michelet, 1986). Structure cristalline du benzoyl-imino-2 triméthyl-1,4,6 dihydro-1,2 pyridine (Rodier, Gillo, Piessard & Le Baut, 1986). Etude spectrale de quelques *N*-(pyridinyl-2) benzamides et structure cristalline du *N*-éthyl *N*-(diméthyl-4,6 pyridinyl-2) benzamide (Rodier, Piessard, Le Baut & Brion, 1987). Synthèse et activités de dérivés de l'amino-2 diméthyl-4,6 pyridine et d'analogues structuraux à

* Le listes des facteurs de structure observés et calculés, des coefficients d'agitation thermique anisotrope, des paramètres des atomes d'hydrogène, des distances C—H et N—H, des distances des atomes aux plans moyens, des distances interatomiques intermoléculaires, et des angles de torsion ont été déposées aux archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 52210: 15 pp.). On peut en obtenir des copies en s'adressant à: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

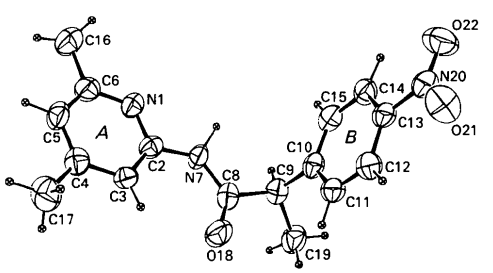

Fig. 1. Vue en perspective de la molécule et numéros attribués à ses atomes.

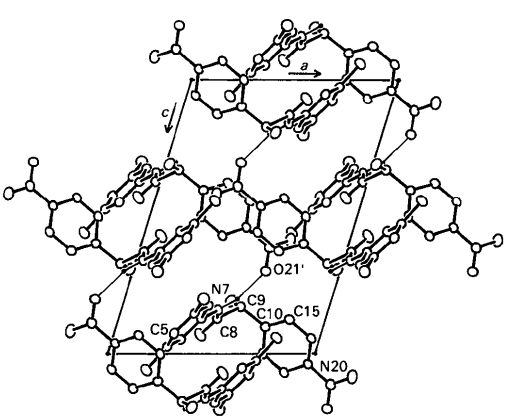

Fig. 2. Vue de la structure selon [010]. Les traits les plus fins représentent la liaison hydrogène N(7)—H(7)···O(21').

Tableau 1. *Coordonnées atomiques relatives, facteurs de température isotropes équivalents et écarts-type*

$B_{éq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab\cos\gamma + \beta_{13}ac\cos\beta + \beta_{23}bc\cos\alpha)$.

	x	y	z	$B_{éq}$(Å^2)
N(1)	0,4136 (3)	0,4976 (3)	0,8243 (2)	3,65 (8)
C(2)	0,3998 (4)	0,4000 (4)	0,8729 (3)	3,3 (1)
C(3)	0,3321 (4)	0,3919 (4)	0,9476 (3)	3,6 (1)
C(4)	0,2756 (4)	0,4922 (4)	0,9741 (3)	3,7 (1)
C(5)	0,2896 (4)	0,5934 (4)	0,9247 (3)	4,1 (1)
C(6)	0,3593 (4)	0,5944 (4)	0,8518 (3)	3,9 (1)
N(7)	0,4669 (3)	0,3058 (3)	0,8401 (2)	3,56 (8)
C(8)	0,4750 (4)	0,1935 (4)	0,8712 (3)	3,8 (1)
C(9)	0,5715 (4)	0,1180 (4)	0,8283 (3)	3,8 (1)
C(10)	0,7188 (4)	0,1252 (3)	0,9017 (3)	3,5 (1)
C(11)	0,7472 (4)	0,0763 (4)	0,9993 (3)	3,7 (1)
C(12)	0,8778 (4)	0,0845 (4)	1,0680 (3)	3,8 (1)
C(13)	0,9803 (4)	0,1424 (3)	1,0381 (3)	3,3 (1)
C(14)	0,9580 (4)	0,1889 (4)	0,9438 (3)	3,8 (1)
C(15)	0,8262 (4)	0,1800 (4)	0,8755 (3)	4,0 (1)
C(16)	0,3817 (5)	0,7036 (4)	0,8002 (4)	5,8 (1)
C(17)	0,2095 (5)	0,4896 (5)	1,0587 (3)	5,7 (1)
O(18)	0,4173 (3)	0,1546 (3)	0,9302 (2)	5,83 (8)
C(19)	0,5164 (5)	−0,0040 (4)	0,8077 (4)	5,3 (1)
N(20)	1,1208 (3)	0,1537 (3)	1,1118 (3)	4,16 (9)
O(21)	1,1391 (3)	0,1191 (3)	1,1985 (2)	5,67 (9)
O(22)	1,2121 (3)	0,1963 (3)	1,0833 (3)	6,8 (1)

Tableau 2. *Longueurs* (Å), *angles des liaisons* (°) *et écarts-type*

N(1)—C(2)	1,334 (5)	C(8)—O(18)	1,210 (6)
N(1)—C(6)	1,344 (6)	C(9)—C(10)	1,522 (5)
C(2)—C(3)	1,386 (7)	C(9)—C(19)	1,507 (6)
C(2)—N(7)	1,418 (5)	C(10)—C(11)	1,399 (6)
C(3)—C(4)	1,383 (6)	C(10)—C(15)	1,384 (6)
C(4)—C(5)	1,375 (6)	C(11)—C(12)	1,370 (5)
C(4)—C(17)	1,494 (7)	C(12)—C(13)	1,383 (6)
C(5)—C(6)	1,375 (7)	C(13)—C(14)	1,353 (6)
C(6)—C(16)	1,492 (7)	C(13)—N(20)	1,476 (5)
N(7)—C(8)	1,358 (5)	C(14)—C(15)	1,379 (5)
N(7)···O(21')	3,074 (5)*	N(20)—O(21)	1,213 (5)
C(8)—C(9)	1,540 (6)	N(20)—O(22)	1,200 (5)

C(2)—N(1)—C(6)	116,9 (4)	C(8)—C(9)—C(19)	111,2 (4)
N(1)—C(2)—C(3)	124,7 (4)	C(10)—C(9)—C(19)	113,9 (3)
N(1)—C(2)—N(7)	111,1 (4)	C(9)—C(10)—C(11)	119,5 (4)
C(3)—C(2)—N(7)	124,2 (4)	C(9)—C(10)—C(15)	122,0 (4)
C(2)—C(3)—C(4)	117,6 (4)	C(11)—C(10)—C(15)	118,5 (3)
C(3)—C(4)—C(5)	118,2 (4)	C(10)—C(11)—C(12)	120,8 (4)
C(3)—C(4)—C(17)	119,7 (4)	C(11)—C(12)—C(13)	118,2 (4)
C(5)—C(4)—C(17)	122,1 (4)	C(12)—C(13)—C(14)	123,0 (3)
C(4)—C(5)—C(6)	120,7 (4)	C(12)—C(13)—N(20)	118,8 (3)
N(1)—C(6)—C(5)	121,9 (4)	C(14)—C(13)—N(20)	118,2 (4)
N(1)—C(6)—C(16)	116,1 (4)	C(13)—C(14)—C(15)	118,2 (4)
C(5)—C(6)—C(16)	122,0 (4)	C(10)—C(15)—C(14)	121,3 (4)
C(2)—N(7)—C(8)	128,5 (4)	C(13)—N(20)—O(21)	118,5 (4)
N(7)—C(8)—C(9)	114,0 (4)	C(13)—N(20)—O(22)	118,6 (3)
N(7)—C(8)—O(18)	124,5 (4)	O(21)—N(20)—O(22)	122,8 (3)
C(9)—C(8)—O(18)	121,5 (4)	N(7)—H(7)···O(21')	164*
C(8)—C(9)—C(10)	108,2 (3)		

* Liaison hydrogène. (i) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$.

polarité antiinflammatoire (Bouhayat, 1981). Synthèse et effets dopaminergiques centraux des *N*-(diméthyl-4,6 pyridinyl-2) benzamides (Bouhayat, Piessard, Le Baut, Sparfel, Petit, Piriou & Welin, 1985).

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Structure of (1*R*,5*S*,8*R*)-8-Methoxy-3,3-dimethyl-2,4,7-trioxabicyclo-[3.3.0]octan-6-one

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Abstract. $C_8H_{12}O_5$, $M_r = 188{\cdot}18$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}164\ (2)$, $b = 9{\cdot}388\ (3)$, $c = 13{\cdot}813\ (3)$ Å, $V = 928{\cdot}9\ (8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}345$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}06$ cm^{-1}, $F(000) = 400$, $T = 90$ K, final $R = 0{\cdot}024$ for 1474 unique observed reflections with $I > 2\sigma(I)$ and 167 variables. The determination was carried out to establish unambiguously the structure of an unexpected reaction by-product. The lactone ring is nearly planar, while the acetonide ring has an envelope conformation with the C atom carrying the *gem*-methyl substituents lying out of plane. The angle between the ring planes is 109·15°. Bond distances and angles are unexceptional.

Experimental. The title compound was isolated as a by-product of oxidation of methyl 2,3-*O*-isopropylidene-β-D-ribofuranoside with pyridinium dichromate. The by-product was obtained after flash chromatography and fractional distillation. The compound was recrystallized as colourless prisms, m.p. 352–353 K (sealed tube), $[\alpha]_D^{25°C} -53{\cdot}3$ ($c = 1$, $CHCl_3$). The X-ray diffraction measurements were made on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. The crystal used was cut to an approximate size 0·34 × 0·31 × 0·39 mm. The temperature at the crystal site was 90 K. Cell parameters were based on a least-squares fit of 25 independent reflections with $28 < 2\theta < 40°$. The intensity data were recorded using the ω-scan technique with a constant scan speed of 2·7° min^{-1}. The crystal orientation was checked every 200 recordings. Three standard reflections were measured every hour. They varied by less than 2%, the variations being irregular with respect to time. The data were corrected for Lp and absorption effects. The latter correction was empirical (Walker & Stewart, 1983); minimum correction factor was 0·94 and maximum 1·05. 1567 independent reflections were recorded ($2\theta < 60°$), h 0–10, k 0–13, l 0–19, 93 reflections with $I < 2{\cdot}0\sigma(I)$ were regarded as unobserved. The structure was solved by *MULTAN*80 (Main *et al.*, 1980) and refined by full-matrix least-squares minimization of $\sum w(\Delta F)^2$, where $w^{-1} = \sigma^2(I)/4\mathrm{Lp}I$, $\sigma^2(I) = \sigma^2(I_c) + 0{\cdot}02(I_c)^2$ and $I_c = I_{\mathrm{count}}$. Non-H atoms were refined with anisotropic and H atoms with isotropic thermal parameters. The final shift-to-e.s.d. ratio was $\leq 0{\cdot}01$. The difference Fourier map after final least-squares

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