

Fig. 3. Vue en perspective de la molécule.

atomes d'hydrogène a conduit à un facteur résiduel de $R = 0,045$. Les facteurs de diffusion atomiques sont ceux des *International Tables for X-ray Crystallography* (1974) pour les atomes de C, N et O et ceux de Stewart, Davidson & Simpson (1965) pour les atomes d'hydrogène. Le schéma de pondération utilisé lors de l'affinement est le suivant: $w^{1/2} = 1$ si $|F_o| < p$ et $w^{1/2} = p/F_o$ si $|F_o| > p$ avec $p = (F_{o\max}^2/10)^{1/2}$.

Le Tableau 1 fournit les coordonnées atomiques et les écarts-type correspondants.* Les longueurs de liaison, les angles de valence et les angles dièdres sont donnés sur la Fig. 2.

Discussion. La molécule synthétisée correspond à la formule (2). Le cycle B hepténique à la forme 'twist-boat' (Bucourt, 1974). Le cycle C heptatriénique est fortement plissé. Cette déformation est susceptible

* Les listes des facteurs de structure et des facteurs d'agitation thermique anisotrope ont été déposées au dépôt d'archives de la British Library Lending Division (Supplementary Publication No. SUP 34312: 29 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.

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Ethyl 3-Nitrato-2-nitro-3-(4-nitrophenyl)propionate

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Abstract. $C_{11}H_{11}N_3O_6$, $M_r = 329.2$, monoclinic, $P2_1/n$, $a = 13.466$ (3), $b = 13.609$ (3), $c = 7.745$ (3) Å, $\beta = 94.13$ (2)°, $U = 1415.7$ Å³; systematic extinctions: $h0l$, $h + l = 2n + 1$; $0k0$, $k = 2n + 1$; $D_c = 1.54$ Mg m⁻³, $Z = 4$; Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 0.13$ mm⁻¹. The propionate group has a staggered conformation about $C_\alpha-C_\beta$. In the *p*-nitrobenzene moiety the NO_2 group is twisted out of the plane of the benzene ring by 15.11° .

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d'entraîner des perturbations dans les propriétés colorimétriques du système heptafulvénique défini par le cycle C et la liaison C(15)–C(7).

Les autres perturbations conformationnelles concernent la jonction des cycles B et C qui est à peu près plane pour la colchicine naturelle et qui ici se coude nettement (environ 50°). La Fig. 3 est une vue en perspective de la molécule.

Par contre les angles existant entre le cycle aromatique A et les trois groupements méthoxy restent sensiblement les mêmes. Ils valent respectivement 67, 56 et 13° pour les méthoxy fixés sur C(1), C(2) et C(3). Les autres angles des groupements avec les cycles hepténiques sont: 79° entre le cycle B et le plan [C(7)–N(27)–C(37)–C(67)]; 47° entre le cycle C et le plan [C(9)–O(19)–C(29)]; 19° entre le cycle C et le plan [C(10)–O(20)–C(30)].

La cohésion cristalline est assurée par un ensemble de contacts de van der Waals.

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Introduction. Transparent cubic crystals of the title compound were supplied by Dr P. G. Taylor. The crystals were moisture sensitive and were mounted under dry nitrogen in glass capillaries. After survey photography, the selected crystal was mounted on a Nonius CAD-4-F, PDP8-controlled kappa geometry diffractometer; cell dimensions and the orientation matrix were obtained by a least-squares method from the setting angles of 25 reflexions.

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The intensities of reflexions with $\sin \theta/\lambda < 0.66 \text{ \AA}^{-1}$ were measured by an $\omega/2\theta$ scan, a variable scan rate and an ω scan angle of $(1.20 + 0.35 \tan \theta)^\circ$. Mo $K\alpha$ radiation was used with a graphite monochromator. Reflexions with $I < 3\sigma(I)$, where $\sigma(I)$ is the e.s.d. based on simple counting statistics, were not included in subsequent calculations. Corrections for Lorentz and polarization effects, but not absorption, were made. 1916 independent structure amplitudes were obtained.

With *MULTAN* 77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977), 22 of the 23 non-hydrogen atoms were located from an *E* map based on the best phase set after weighted, multi-solution tangent refinement (Germain, Main & Woolfson, 1971). 250 phases with $E > 1.42$ were used in this refinement. Refinement of the atomic coordinates and isotropic temperature factors, followed by a difference synthesis, revealed the last atom. Refinement was by a least-squares method with a large-block approximation to the normal matrix: one block was calculated from the derivatives of the positional parameters, and one from those of the corresponding temperature factors and the scale factor. All non-hydrogen atoms had anisotropic temperature factors. The H atoms were located from

Table 1. Fractional atomic coordinates with e.s.d.'s in parentheses

	<i>x</i>	<i>y</i>	<i>z</i>
O(1)	0.4943 (3)	0.9059 (3)	0.0712 (5)
O(2)	0.4185 (2)	0.8507 (2)	0.2871 (5)
O(3)	0.8094 (1)	1.0080 (1)	0.8437 (2)
O(4)	0.7636 (1)	1.1246 (2)	1.0117 (2)
O(5)	0.8064 (2)	1.1650 (1)	0.7581 (3)
O(6)	1.0117 (1)	0.8566 (2)	0.9294 (3)
O(7)	1.0317 (1)	0.9919 (1)	0.7918 (3)
O(8)	1.0251 (1)	0.8377 (2)	0.5198 (3)
O(9)	0.8651 (1)	0.8191 (1)	0.4274 (2)
N(1)	0.4884 (2)	0.8903 (2)	0.2263 (5)
N(2)	0.7915 (1)	1.1095 (1)	0.8717 (3)
N(3)	0.9876 (1)	0.9162 (2)	0.8173 (3)
C(1)	0.5749 (2)	0.9190 (2)	0.3439 (4)
C(2)	0.5821 (2)	0.8816 (2)	0.5081 (4)
C(3)	0.6653 (2)	0.9042 (2)	0.6155 (3)
C(4)	0.7391 (1)	0.9641 (2)	0.5560 (3)
C(5)	0.7287 (2)	1.0037 (2)	0.3909 (3)
C(6)	0.6453 (2)	0.9797 (2)	0.2819 (3)
C(7)	0.8334 (1)	0.9835 (2)	0.6687 (3)
C(8)	0.8976 (1)	0.8912 (2)	0.6983 (3)
C(9)	0.9385 (2)	0.8468 (2)	0.5365 (3)
C(10)	0.8905 (3)	0.7694 (2)	0.2689 (3)
C(11)	0.8023 (3)	0.7754 (3)	0.1436 (4)
H(20)	0.534 (1)	0.838 (2)	0.544 (1)
H(30)	0.672 (1)	0.878 (2)	0.733 (3)
H(50)	0.778 (2)	1.045 (2)	0.352 (2)
H(60)	0.6397 (8)	1.002 (1)	0.162 (3)
H(70)	0.872 (1)	1.037 (1)	0.620 (1)
H(80)	0.860 (1)	0.842 (1)	0.753 (2)
H(100)	0.906 (2)	0.700 (2)	0.294 (2)
H(101)	0.948 (2)	0.803 (2)	0.221 (1)
H(110)	0.748 (2)	0.744 (2)	0.197 (3)
H(111)	0.818 (1)	0.737 (2)	0.044 (3)
H(112)	0.794 (2)	0.843 (2)	0.112 (4)

difference syntheses and included in the refinement with isotropic temperature factors. All bond lengths and angles involving H atoms were constrained (Waser, 1963; Rollett, 1969), and the difference in mean-square displacement along the bond direction of the two atoms (C and H) forming each bond was constrained to be zero with an e.s.d. of 0.001 \AA^2 . In the final stages, each

Table 2. Interatomic distances (Å) and bond angles (°)

O(1)—N(1)	1.228 (5)	N(2)—O(4)	1.191 (2)
O(2)—N(1)	1.209 (4)	N(2)—O(5)	1.188 (3)
N(1)—C(1)	1.479 (3)	C(7)—C(8)	1.534 (3)
C(1)—C(2)	1.366 (4)	C(8)—N(3)	1.508 (3)
C(2)—C(3)	1.382 (3)	N(3)—O(6)	1.215 (3)
C(3)—C(4)	1.390 (3)	N(3)—O(7)	1.212 (3)
C(4)—C(5)	1.385 (3)	C(8)—C(9)	1.529 (3)
C(5)—C(6)	1.395 (3)	C(9)—O(8)	1.189 (3)
C(6)—C(1)	1.370 (4)	C(9)—O(9)	1.309 (3)
C(4)—C(7)	1.511 (3)	O(9)—C(10)	1.463 (3)
C(7)—O(3)	1.454 (3)	C(10)—C(11)	1.481 (5)
O(3)—N(2)	1.422 (2)		
O(1)—N(1)—O(2)	124.4 (3)	C(7)—O(3)—N(2)	114.6 (2)
O(1)—N(1)—C(1)	117.0 (3)	O(3)—N(2)—O(4)	112.0 (2)
O(2)—N(1)—C(1)	118.6 (3)	O(3)—N(2)—O(5)	117.8 (2)
N(1)—C(1)—C(2)	118.3 (3)	O(4)—N(2)—O(5)	130.3 (2)
N(1)—C(1)—C(6)	118.5 (3)	C(7)—C(8)—N(3)	109.0 (2)
C(2)—C(1)—C(6)	123.1 (2)	C(7)—C(8)—C(9)	115.7 (2)
C(1)—C(2)—C(3)	118.5 (2)	N(3)—C(8)—C(9)	105.6 (2)
C(2)—C(3)—C(4)	119.9 (2)	O(6)—N(3)—O(7)	125.1 (2)
C(3)—C(4)—C(5)	120.5 (2)	O(6)—N(3)—C(8)	116.6 (2)
C(3)—C(4)—C(7)	120.1 (2)	O(7)—N(3)—C(8)	118.3 (2)
C(5)—C(4)—C(7)	119.4 (2)	C(8)—C(9)—O(8)	123.0 (2)
C(4)—C(5)—C(6)	119.4 (2)	C(8)—C(9)—O(9)	110.1 (2)
C(5)—C(6)—C(1)	118.5 (2)	O(8)—C(9)—O(9)	126.9 (2)
C(4)—C(7)—O(3)	110.2 (2)	C(9)—O(9)—C(10)	117.6 (2)
C(4)—C(7)—C(8)	112.6 (2)	O(9)—C(10)—C(11)	107.4 (3)
O(3)—C(7)—C(8)	102.0 (2)		

Table 3. Equations of least-squares planes and, in square brackets, distances (Å) of atoms from the planes

The equations are referred to the crystal axes and *x, y, z* are fractional coordinates.

Plane 1: ring C(1) to C(6)

$$6.725x - 10.868y - 2.874z = -7.119$$

[C(1) 0.010, C(2) -0.009, C(3) -0.002, C(4) 0.013, C(5) -0.012, C(6) 0.001, N(1)* 0.077, O(1)* 0.393, O(2)* -0.138]

Plane 2: N(1), O(1), O(2)

$$6.003x - 12.112y - 0.986z = -8.075$$

Plane 3: C(8), N(3), O(6), O(7)

$$-8.435x + 6.459y + 5.127z = 1.767$$

[C(8) -0.003, N(3) 0.010, O(6) -0.003, O(7) -0.004]

Plane 4: O(3), N(2), O(4), O(5)

$$12.426x + 1.514y + 2.336z = 13.553$$

[O(3) 0.001, N(2) -0.003, O(4) 0.001, O(5) 0.001]

Plane 5: C(8), C(9), O(8), O(9)

$$-0.483x - 12.291y + 3.325z = -9.068$$

[C(8) 0.003, C(9) -0.010, O(8) 0.004, O(9) 0.003]

* Atoms not included in the plane calculation.

Table 4. Torsion angles ($^{\circ}$)

The torsion angle about the bond $J-K$ is the angle the bond $K-L$ is rotated from the IJK plane. It is positive when, on looking from IJ to KL , the rotation is clockwise.

O(3)-C(7)-C(8)-C(9)	-178.48	H(70)-C(7)-C(8)-N(3)	57.70	O(1)-N(1)-C(1)-C(2)	-164.02
O(3)-C(7)-C(8)-N(3)	-59.69	H(70)-C(7)-C(8)-H(80)	176.09	O(1)-N(1)-C(1)-C(6)	14.43
O(3)-C(7)-C(8)-H(80)	58.70	C(7)-C(8)-C(9)-O(8)	120.15	O(2)-N(1)-C(1)-C(2)	13.66
C(4)-C(7)-C(8)-C(9)	63.42	C(7)-C(8)-C(9)-O(9)	-61.71	O(2)-N(1)-C(1)-C(6)	-167.89
C(4)-C(7)-C(8)-N(3)	-177.79	N(3)-C(8)-C(9)-O(8)	-0.52		
C(4)-C(7)-C(8)-H(80)	-59.40	N(3)-C(8)-C(9)-O(9)	177.62		
H(70)-C(7)-C(8)-C(9)	-61.09				

reflexion was assigned a weight $w = 1/\sum_{r=1}^n A_r T_r^*(X)$, where n is the number of coefficients, A_r , for a Chebyshev series, T_r^* is the polynomial function and X is $|F_o|/|F_o(\text{max.})|$. Two coefficients, A_r , were used with values 229.86 and 227.22 (Rollett, 1965), and the final R_w was 0.057 for 1916 reflexions.

All calculations were performed on the Oxford University ICL 1906A and 2980 computers with the Oxford *CRYSTALS* package (Carruthers, 1978). Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

The final atomic coordinates are given in Table 1.* Table 2 lists bond distances and angles for the non-hydrogen atoms, with e.s.d.'s calculated from the variance-covariance matrix. Equations of molecular planes are given in Table 3 and torsion angles in Table 4.

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34317 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

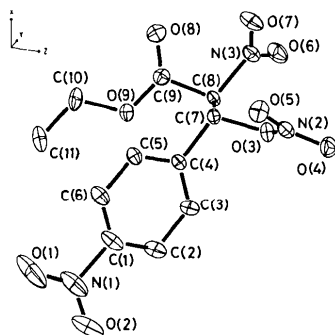


Fig. 1. The molecular structure of ethyl 3-nitrato-2-nitro-3-(4-nitrophenyl)propionate with 50% probability ellipsoids for the non-hydrogen atoms.

Discussion. The X-ray analysis of this nitration product of ethyl *p*-nitrocinnamate was undertaken to determine the stereochemistry about $C_{\alpha}-C_{\beta}$ [C(7)-C(8)] in the propionate moiety. The conformation of the molecule and the atom numbering are shown in Fig. 1.

In the propionate group there is a staggered arrangement about C(7)-C(8), as shown by the torsion angles. Bond distances and angles are normal and compare well with those found in 3-(*p*-chlorophenyl)propionic acid (Glusker, Zacharias & Carrell, 1975), where there is also a staggered conformation.

The benzene ring is planar (plane 1, Table 3) and the C-C distances are normal. The wide C(2)-C(1)-C(6) [$123.1(2)^{\circ}$] and small C(1)-C(2)-C(3) [$118.5(2)^{\circ}$] and C(5)-C(6)-C(1) [$118.5(2)^{\circ}$] angles are found also in other rings with a NO_2 group in the *para* position, for example in *p*-nitrobenzoic acid (Tavale & Pant, 1971), 3,5-dimethyl-4-nitrobenzoic acid (Aprea, Smith-Verdier & Garcia-Blanco, 1973) and *p*- $\text{NO}_2-\text{C}_6\text{H}_4-\text{C}(\text{Cl})=\text{NOH}$ (Declercq, Germain & Van Meerssche, 1975). The NO_2 group is twisted about N(1)-C(1) away from the plane of the ring by 15.11° (Table 4), similar to that found in *p*-nitrobenzoic acid.

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3 β ,28-Diacetoxy-18 β ,19 β -epoxylupane

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Abstract. C₃₄H₅₄O₅, orthorhombic, *P*2₁2₁2₁, *a* = 8.088 (1), *b* = 11.467 (2), *c* = 33.321 (8) Å (at 295 K), *V* = 3090.4 Å³, *Z* = 4, *M_r* = 542.8, *D_c* = 1.17 Mg m⁻³, *F*(000) = 1192, $\lambda(\text{Mo } K\alpha) = 0.7107 \text{ \AA}$, $\mu(\text{Mo } K\alpha) = 0.079 \text{ mm}^{-1}$. *R* = 0.035 for 1765 diffractometer-measured reflections. The structure confirms the β -epoxidation of a pentacyclic triterpene at the *E* ring.

Introduction. 3 β ,28-Diacetoxy-18 β ,19 β -epoxylupane is a peracid epoxidation product of a pentacyclic triterpene belonging to the lupane group (Suokas, 1977). Epoxidation of triterpenes usually occurs by attack from the α -side due to steric hindrance of methyl groups on the β -side. However, in this case spectral data indicated that peracid oxidation produced a different epoxide from that obtained by ozone treatment (Suokas & Hase, 1977*a*). Further experiments showed that this mechanism was not due to the directive effect of the C(28) acetoxy group on the β -side (Suokas & Hase, 1977*b*). Thus, a crystallographic study of the title compound was initiated to establish the structures of the epoxides. A further interesting feature of the title compound was the fact that triterpenes, especially the pentacyclic ones, have been relatively seldom studied by X-ray methods.

The systematic absences in the diffraction pattern indicated the space group *P*2₁2₁2₁ (No. 19), and the cell parameters were obtained from a least-squares refinement of the angular settings of 25 reflections with a Syntex *P*2₁ (Fortran version) diffractometer. All reflections up to $2\theta = 45^\circ$ were measured on the same diffractometer with graphite-monochromatized Mo *K* α radiation by the θ - 2θ method and at a variable scan rate of 1 to 15° min⁻¹. Using the criterion $I > 2\sigma(I)$, 1787 reflections were considered as 'observed' and were

Table 1. Final atomic coordinates for the nonhydrogen atoms ($\times 10^4$)

	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	10766 (4)	4377 (3)	7594 (1)
C(2)	11482 (5)	4307 (3)	8021 (1)
C(3)	10626 (5)	5159 (3)	8293 (1)
C(4)	8739 (5)	5025 (3)	8326 (1)
C(5)	8072 (4)	5037 (3)	7885 (1)
C(6)	6183 (4)	4998 (4)	7857 (1)
C(7)	5618 (4)	5387 (3)	7439 (1)
C(8)	6313 (4)	4627 (3)	7095 (1)
C(9)	8233 (4)	4543 (3)	7151 (1)
C(10)	8866 (4)	4178 (3)	7578 (1)
C(11)	9039 (4)	3874 (3)	6805 (1)
C(12)	8641 (4)	4413 (3)	6398 (1)
C(13)	6764 (4)	4477 (3)	6332 (1)
C(14)	5902 (4)	5213 (3)	6669 (1)
C(15)	3999 (4)	5211 (4)	6596 (1)
C(16)	3511 (5)	5589 (3)	6168 (1)
C(17)	4336 (5)	4807 (3)	5844 (1)
C(18)	6213 (4)	4823 (3)	5918 (1)
C(19)	7003 (5)	5520 (4)	5588 (1)
C(20)	8720 (6)	6031 (4)	5556 (1)
C(21)	5639 (6)	6020 (4)	5327 (1)
C(22)	4095 (5)	5318 (4)	5422 (1)
C(23)	8074 (6)	6132 (4)	8542 (1)
C(24)	8244 (6)	3945 (4)	8573 (1)
C(25)	8561 (6)	2873 (3)	7672 (1)
C(26)	5478 (5)	3404 (3)	7122 (1)
C(27)	6492 (5)	6489 (3)	6650 (1)
C(28)	3777 (5)	3539 (4)	5865 (1)
C(29)	8788 (8)	7304 (5)	5676 (2)
C(30)	9363 (6)	5894 (5)	5127 (1)
C(31)	12673 (5)	5653 (4)	8792 (1)
C(32)	13279 (7)	5398 (5)	9201 (1)
C(33)	1846 (6)	2445 (4)	5495 (1)
C(34)	160 (7)	2424 (5)	5317 (2)
O(35)	6999 (4)	4238 (2)	5578 (1)
O(36)	11318 (3)	5032 (2)	8699 (1)
O(37)	2146 (3)	3440 (2)	5695 (1)
O(38)	13301 (4)	6325 (3)	8564 (1)
O(39)	2806 (4)	1670 (3)	5470 (1)