| C18 | −0.2428 (2) | −0.0784 (1) | 0.0190 (2) | 0.0518 (4) |
| C19 | −0.1489 (2) | 0.3518 (1) | 0.0759 (1) | 0.0490 (4) |
| O20 | −0.1926 (3) | 0.3904 (1) | −0.0076 (2) | 0.1249 (9) |
| O21 | −0.0859 (1) | 0.3945 (1) | 0.1654 (1) | 0.0526 (3) |
| C22 | −0.0802 (3) | 0.4914 (1) | 0.1615 (2) | 0.0610 (5) |
| C23 | 0.0134 (3) | 0.5231 (1) | 0.2643 (2) | 0.0641 (5) |

Table 2. *Selected geometric parameters* (Å, °)

| O1—C9 | 1.376 (2) | C8—C9 | 1.378 (2) |
| O1—C2 | 1.394 (2) | C8—C18 | 1.509 (2) |
| C2—O11 | 1.202 (2) | C9—C10 | 1.408 (2) |
| C2—C3 | 1.451 (2) | C12—C13 | 1.519 (3) |
| C3—C4 | 1.369 (2) | C13—C14 | 1.503 (3) |
| C3—C19 | 1.472 (2) | C14—N15 | 1.459 (2) |
| C4—C10 | 1.408 (2) | N15—C16 | 1.463 (2) |
| C5—C6 | 1.366 (2) | C16—C17 | 1.500 (3) |
| C5—C10 | 1.411 (2) | C17—C18 | 1.522 (2) |
| C6—C7 | 1.436 (2) | C19—O20 | 1.188 (2) |
| C6—C12 | 1.505 (2) | C19—O21 | 1.322 (2) |
| C7—N15 | 1.365 (2) | O21—C22 | 1.450 (2) |
| C7—C8 | 1.410 (2) | C22—C23 | 1.487 (3) |
| C9—O1—C2 | 123.6 (1) | C14—N15—C16 | 114.5 (1) |
| O11—C2—O1 | 115.1 (1) | N15—C16—C17 | 112.1 (2) |
| O11—C2—C3 | 128.6 (2) | C16—C17—C18 | 110.2 (2) |
| O1—C2—C3 | 116.3 (1) | C8—C18—C17 | 109.3 (1) |
| C6—C12—C13 | 109.2 (2) | O20—C19—O21 | 121.7 (2) |
| C14—C13—C12 | 109.4 (2) | O20—C19—C3 | 125.1 (2) |
| N15—C14—C13 | 112.4 (1) | O21—C19—C3 | 113.0 (1) |
| C7—N15—C14 | 123.4 (1) | C19—O21—C22 | 117.9 (1) |
| C7—N15—C16 | 122.0 (1) | O21—C22—C23 | 108.0 (2) |
| C12—C6—C7—N15 | −0.8 (2) | C8—C7—N15—C16 | −6.8 (2) |
| N15—C7—C8—C18 | 3.2 (2) | C13—C14—N15—C7 | −13.7 (3) |
| C7—C6—C12—C13 | 36.3 (2) | C7—N15—C16—C17 | −22.2 (2) |
| C6—C12—C13—C14 | −59.9 (2) | N15—C16—C17—C18 | 53.1 (2) |
| C12—C13—C14—N15 | 49.3 (2) | C7—C8—C18—C17 | 28.0 (2) |
| C6—C7—N15—C14 | −11.7 (3) | C16—C17—C18—C8 | −55.0 (2) |

The structure was solved in the space group $P2_1$ and refined in $P2_1/n$. All the H atoms were located from difference Fourier maps and refined isotropically. *PARST* (Nardelli, 1983*b*) was used for geometrical calculations and *SHELXTL/PC* (Sheldrick, 1990) for molecular graphics.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL93*.

## References

Chinnakali, K., Selladurai, S., Sivakumar, K., Subramanian, K. & Natarajan, S. (1990). *Acta Cryst.* C46, 837–839.
Chinnakali, K., Sivakumar, K. & Natarajan, S. (1990). *Acta Cryst.* C46, 669–671.
Fait, J. (1991). *XSCANS Users Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Low, J. N. & Wilson, C. C. (1984). *Acta Cryst.* C40, 1030–1032.
Nardelli, M. (1983*a*). *Acta Cryst.* C39, 1141–1142.
Nardelli, M. (1983*b*). *Comput. Chem.* 7, 95–98.
Ravikumar, K., Rajan, S. S., Sivakumar, K. & Natarajan, S. (1989). *Acta Cryst.* C44, 1996–1999.
Reynolds, G. A. & Drexhage, K. H. (1975). *Opt. Commun.* 13, 222–225.

Sahyun, M. R. V. & Sharma, D. K. (1992). *Chem. Phys. Lett.* 189, 571–576.
Sheldrick, G. M. (1990). *Acta Cryst.* A46, 467–473.
Sheldrick, G. M. (1990). *SHELXTL/PC Users Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.
Skrzat, Z. & Roszak, A. (1986). *Acta Cryst.* C42, 1194–1196.

# 3-Hydroxyimino-5α,13α,14β,17α-lanosta-8,24-dien-20-oic Acid

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## Abstract

Rings *B* and *C* in the title compound, $C_{30}H_{47}NO_3$, have a common double bond and adopt an envelope shape, whereas ring *D* assumes a half-chair conformation. Crystal packing is established by intermolecular hydrogen bonds forming infinite helices of the molecules.

## Comment

'Elemi' acids isolated from *Manila elemi* resins may exist in either 3-hydroxy or 3-oxo forms. Ruzicka and co-workers (Ruzicka & Häusermann, 1942; Ruzicka, Rey & Spillmann, 1942; Ruzicka, Rey, Spillmann & Baumgartner, 1943) systematically elucidated the relationships between the tetracyclic triterpenes (*e.g.* squalene, lanosterol) and, among others, derived the chemical structures of α-elemolic and β-elemonic acids. These natural products may be used as raw materials for the semisynthesis of some biologically active steroids. The crystal structure of the title compound, (I), *i.e.* the oxime of β-elemonic acid, is reported in this paper.

(I)
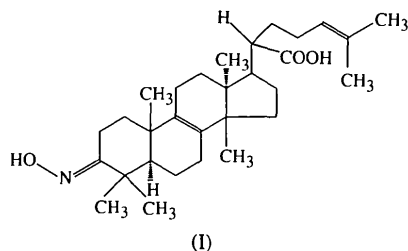
Ring *A* with the oxime group at C3 assumes a chair conformation, whereas ring *B*, which shares a double bond with ring *C*, assumes an envelope shape with C5 on the flap [puckering parameters (Cremer & Pople, 1975): $[Q = 0.521\,(1)\,\text{Å}$, $\varphi = 5.8\,(3)°$, $\theta = 46.0\,(2)°$, with a lowest asymmetry factor (Kálmán, Czugler & Simon, 1982) of $fC_s(\text{C5}) = 0.03\,(6)\,\text{Å}]$. Similarly ring *C* exhibits an envelope form with C13 on the flap $[Q = 0.524\,(2)\,\text{Å}$, $\varphi = 108.2\,(3)°$, $\theta = 53.6\,(2)°$, $fC_s(\text{C13}) = 0.09\,(16)\,\text{Å}]$. The five-membered ring *D*, which is *trans*-fused to ring *C*, adopts a half-chair form with a pseudo-twofold symmetry axis running through C16 and the midpoint of the C13—C14 bond $[Q = 0.475\,(2)\,\text{Å}$, $\varphi = 17.7\,(3)°$, $fC_2(\text{C16}) = 0.00\,(1)\,\text{Å}]$.

The C17 side chain may be partitioned into three planar groups, each formed by four non-H atoms, C17—C20—C21—C22, C22—C23—C24—C25 and C22—C23—C24—C26. The planar carboxyl group [maximum deviation $0.005\,(1)\,\text{Å}$] is perpendicular $[88.9\,(1)°]$ to the C17—C20—C21—C22 group, while the best plane of the terminal isoprenyl moiety [maximum deviation $0.040\,(1)\,\text{Å}$] makes an angle of $73.8\,(1)°$ with the plane of C17—C20—C21—C22 [torsion angle: $173.4\,(3)°$].

The helical array of the half-moon-shaped molecules is maintained by intermolecular hydrogen-bond pairs at both ends of the molecules (Table 3). Each oxime group forms two hydrogen bonds with the carboxyl
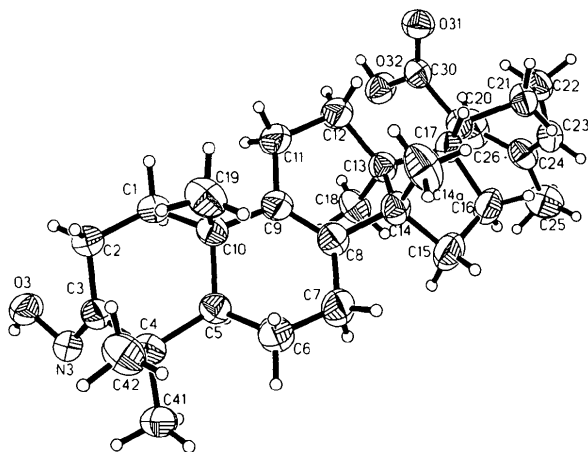
moiety (Fig. 2) pertaining to the symmetry-equivalent molecule translated by $y = 0.5$ along the screw axis. In these hydrogen-bond pairs both =N—OH and —COOH groups act simultaneously as donor and acceptor.
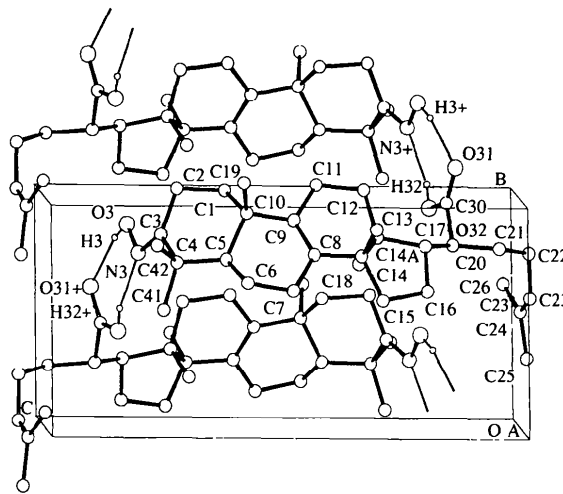


Fig. 2. Close packing showing hydrogen-bond pairs at both ends of the molecules.

## Experimental

The title compound was prepared by the method of Clarke (1975) and crystallized from a mixture of solvents EtOH–BuAc (1:1 *v/v*). M.p. 491–492 K.

*Crystal data*

| | |
|---|---|
| $C_{30}H_{47}NO_3$ | Cu $K\alpha$ radiation |
| $M_r = 469.69$ | $\lambda = 1.5418\,\text{Å}$ |
| Monoclinic | Cell parameters from 25 |
| $P2_1$ | reflections |
| $a = 12.995\,(1)\,\text{Å}$ | $\theta = 43.96\text{–}46.16°$ |
| $b = 7.161\,(1)\,\text{Å}$ | $\mu = 0.547\,\text{mm}^{-1}$ |
| $c = 16.187\,(1)\,\text{Å}$ | $T = 293\,(2)\,\text{K}$ |
| $\beta = 112.37\,(1)°$ | Transparent block |
| $V = 1393.0\,(2)\,\text{Å}^3$ | $0.70 \times 0.55 \times 0.40$ mm |
| $Z = 2$ | Colourless |
| $D_x = 1.120\,\text{Mg m}^{-3}$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 | $R_{int} = 0.0081$ |
| diffractometer | $\theta_{max} = 75.57°$ |
| $\omega/2\theta$ scans | $h = -16 \rightarrow 16$ |
| Absorption correction: | $k = -8 \rightarrow 8$ |
| not applied | $l = -20 \rightarrow 20$ |
| 6630 measured reflections | 3 standard reflections |
| 5757 independent reflections | frequency: 60 min |
| 5684 observed reflections | intensity decay: none |
| $[I > 2\sigma(I)]$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Extinction correction: |
| $R[F^2 > 2\sigma(F^2)] = 0.0337$ | *SHELXL93* (Sheldrick, |
| $wR(F^2) = 0.0968$ | 1994) |

Fig. 1. A perspective view of the molecule with atom-numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

<div style="text-align:center">C₃₀H₄₇NO₃</div>

$S = 1.064$
5757 reflections
316 parameters
H-atom parameters refined
using a riding model
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.1052P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.012$
$\Delta\rho_{max} = 0.204$ e Å⁻³
$\Delta\rho_{min} = -0.147$ e Å⁻³

Extinction coefficient:
0.0048 (5)
Atomic scattering factors
from *International Tables
for Crystallography* (1992,
Vol. C, Tables 4.2.6.8 and
6.1.1.4)
Absolute configuration:
Flack (1983), h =
0.02 (16)

## Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

| | x | y | z | $U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.22063 (11) | 1.0000 | 0.60864 (9) | 0.0451 (3) |
| C2 | 0.27744 (13) | 1.0112 (3) | 0.71077 (9) | 0.0496 (3) |
| C3 | 0.30450 (11) | 0.8164 (3) | 0.74582 (8) | 0.0426 (3) |
| N3 | 0.25738 (10) | 0.7344 (3) | 0.79196 (7) | 0.0453 (2) |
| O3 | 0.18243 (11) | 0.8491 (2) | 0.81233 (8) | 0.0594 (3) |
| C4 | 0.38394 (11) | 0.7060 (3) | 0.71550 (9) | 0.0444 (3) |
| C41 | 0.38977 (14) | 0.5009 (3) | 0.74442 (10) | 0.0563 (3) |
| C42 | 0.50272 (12) | 0.7877 (4) | 0.76061 (11) | 0.0637 (4) |
| C5 | 0.33519 (10) | 0.7151 (3) | 0.61076 (8) | 0.0405 (2) |
| C6 | 0.40827 (12) | 0.6202 (3) | 0.56869 (10) | 0.0564 (4) |
| C7 | 0.34091 (13) | 0.5824 (3) | 0.47061 (10) | 0.0601 (4) |
| C8 | 0.26028 (10) | 0.7305 (3) | 0.42310 (8) | 0.0445 (3) |
| C9 | 0.23237 (11) | 0.8747 (3) | 0.46507 (9) | 0.0452 (3) |
| C10 | 0.29527 (10) | 0.9090 (3) | 0.56565 (8) | 0.0406 (3) |
| C11 | 0.14680 (13) | 1.0188 (3) | 0.41379 (9) | 0.0536 (3) |
| C12 | 0.08790 (13) | 0.9941 (3) | 0.31216 (9) | 0.0523 (3) |
| C13 | 0.08971 (10) | 0.7903 (3) | 0.28431 (8) | 0.0393 (3) |
| C14 | 0.21247 (10) | 0.7217 (3) | 0.32213 (9) | 0.0460 (3) |
| C14A | 0.29067 (13) | 0.8416 (4) | 0.29173 (11) | 0.0692 (5) |
| C15 | 0.2020 (2) | 0.5302 (3) | 0.27737 (11) | 0.0675 (5) |
| C16 | 0.11439 (13) | 0.5626 (3) | 0.18174 (10) | 0.0576 (4) |
| C17 | 0.05567 (10) | 0.7511 (3) | 0.18297 (8) | 0.0420 (3) |
| C18 | 0.01816 (12) | 0.6722 (3) | 0.32160 (9) | 0.0526 (3) |
| C19 | 0.38958 (13) | 1.0466 (3) | 0.57331 (11) | 0.0609 (4) |
| C20 | -0.06877 (10) | 0.7441 (3) | 0.12138 (8) | 0.0418 (3) |
| C21 | -0.08122 (12) | 0.7299 (3) | 0.02281 (8) | 0.0499 (3) |
| C22 | -0.20097 (14) | 0.6998 (3) | -0.04353 (9) | 0.0565 (4) |
| C23 | -0.24002 (12) | 0.5037 (3) | -0.04643 (9) | 0.0500 (3) |
| C24 | -0.32470 (12) | 0.4382 (3) | -0.02876 (9) | 0.0543 (3) |
| C25 | -0.3542 (2) | 0.2333 (4) | -0.04188 (14) | 0.0766 (5) |
| C26 | -0.3960 (2) | 0.5518 (4) | 0.00540 (13) | 0.0788 (6) |
| C30 | -0.12621 (10) | 0.9203 (3) | 0.13182 (8) | 0.0424 (3) |
| O31 | -0.10347 (9) | 1.0745 (2) | 0.11244 (7) | 0.0544 (2) |
| O32 | -0.20319 (9) | 0.8921 (2) | 0.16563 (7) | 0.0530 (2) |

## Table 2. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.535 (2) | C12—C13 | 1.530 (2) |
| C1—C10 | 1.537 (2) | C13—C18 | 1.540 (2) |
| C2—C3 | 1.497 (2) | C13—C17 | 1.553 (2) |
| C3—N3 | 1.275 (2) | C13—C14 | 1.555 (2) |
| C3—C4 | 1.522 (2) | C14—C15 | 1.532 (2) |
| N3—O3 | 1.406 (2) | C14—C14A | 1.547 (2) |
| C4—C41 | 1.535 (2) | C15—C16 | 1.551 (2) |
| C4—C42 | 1.549 (2) | C16—C17 | 1.554 (2) |
| C4—C5 | 1.569 (2) | C17—C20 | 1.544 (2) |
| C5—C6 | 1.523 (2) | C20—C30 | 1.508 (2) |
| C5—C10 | 1.563 (2) | C20—C21 | 1.545 (2) |
| C6—C7 | 1.517 (2) | C21—C22 | 1.533 (2) |
| C7—C8 | 1.484 (2) | C22—C23 | 1.488 (2) |
| C8—C9 | 1.359 (2) | C23—C24 | 1.324 (2) |
| C8—C14 | 1.513 (2) | C24—C26 | 1.488 (2) |
| C9—C11 | 1.512 (2) | C24—C25 | 1.512 (3) |
| C9—C10 | 1.538 (2) | C30—O31 | 1.215 (2) |
| C10—C19 | 1.540 (2) | C30—O32 | 1.326 (2) |
| C11—C12 | 1.538 (2) | | |

| | | | |
|---|---|---|---|
| C2—C1—C10 | 112.5 (1) | C13—C12—C11 | 111.6 (1) |
| C3—C2—C1 | 107.9 (1) | C12—C13—C18 | 109.2 (1) |
| N3—C3—C2 | 123.7 (1) | C12—C13—C17 | 117.0 (1) |
| N3—C3—C4 | 119.1 (1) | C18—C13—C17 | 110.0 (1) |
| C2—C3—C4 | 116.9 (1) | C12—C13—C14 | 108.3 (1) |
| C3—N3—O3 | 113.5 (1) | C18—C13—C14 | 110.7 (1) |
| C3—C4—C41 | 111.1 (1) | C17—C13—C14 | 101.3 (1) |
| C3—C4—C42 | 109.2 (1) | C8—C14—C15 | 118.3 (1) |
| C41—C4—C42 | 107.0 (1) | C8—C14—C14A | 105.7 (1) |
| C3—C4—C5 | 106.4 (1) | C15—C14—C14A | 107.3 (1) |
| C41—C4—C5 | 108.8 (1) | C8—C14—C13 | 110.5 (1) |
| C42—C4—C5 | 114.5 (1) | C15—C14—C13 | 101.7 (1) |
| C6—C5—C10 | 109.9 (1) | C14A—C14—C13 | 113.5 (1) |
| C6—C5—C4 | 113.6 (1) | C14—C15—C16 | 103.8 (1) |
| C10—C5—C4 | 117.9 (1) | C15—C16—C17 | 107.1 (1) |
| C7—C6—C5 | 109.8 (1) | C20—C17—C13 | 119.7 (1) |
| C8—C7—C6 | 115.3 (1) | C20—C17—C16 | 111.2 (1) |
| C9—C8—C7 | 123.7 (1) | C13—C17—C16 | 103.1 (1) |
| C9—C8—C14 | 119.6 (1) | C30—C20—C21 | 108.0 (1) |
| C7—C8—C14 | 116.7 (1) | C30—C20—C17 | 110.0 (1) |
| C8—C9—C11 | 121.6 (1) | C21—C20—C17 | 110.0 (1) |
| C8—C9—C10 | 121.0 (1) | C22—C21—C20 | 114.4 (1) |
| C11—C9—C10 | 117.1 (1) | C23—C22—C21 | 113.5 (1) |
| C9—C10—C1 | 112.0 (1) | C24—C23—C22 | 129.0 (2) |
| C9—C10—C19 | 105.9 (1) | C23—C24—C26 | 125.0 (2) |
| C1—C10—C19 | 108.2 (1) | C23—C24—C25 | 120.1 (2) |
| C9—C10—C5 | 107.5 (1) | C26—C24—C25 | 115.0 (2) |
| C1—C10—C5 | 108.6 (1) | O31—C30—O32 | 122.8 (1) |
| C19—C10—C5 | 114.7 (1) | O31—C30—C20 | 123.5 (1) |
| C9—C11—C12 | 118.1 (1) | O32—C30—C20 | 113.7 (1) |

## Table 3. *Hydrogen-bonding geometry (Å, °)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| O3—H3···O31ⁱ | 0.81 (2) | 1.93 (2) | 2.712 (3) | 161 (1) |
| O32—H32···N3ⁱⁱ | 0.82 (2) | 1.92 (2) | 2.710 (3) | 161 (1) |

Symmetry codes: (i) $-x, y - \frac{1}{2}, 1 - z$; (ii) $-x, \frac{1}{2} + y, 1 - z$.

Data collection: Enraf–Nonius *EXPRESS* (Enraf–Nonius, 1992). Cell refinement: Enraf–Nonius *EXPRESS*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994).

## References

Clarke, T. H. (1975). *A Handbook of Organic Analysis*, edited by E. Arnold, 5th edition, p. 76. Belfast Univ. Press.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Enraf–Nonius (1992). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Kálmán, A., Czugler, M. & Simon, K. (1982). *Molecular Structure and Biological Activity*, edited by J. F. Griffin & W. L. Duax, pp. 367–376. New York: Elsevier Biomedical.
Ruzicka, L. & Häusermann, H. (1942). *Helv. Chim. Acta*, **25**, 439–457.
Ruzicka, L., Rey, E. & Spillmann, M. (1942). *Helv. Chim. Acta*, **25**, 1375–1402.
Ruzicka, L., Rey, E., Spillmann, M. & Baumgartner, H. (1943). *Helv. Chim. Acta*, **26**, 1638–1658.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1994). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany