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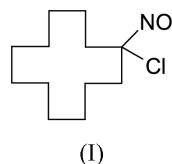
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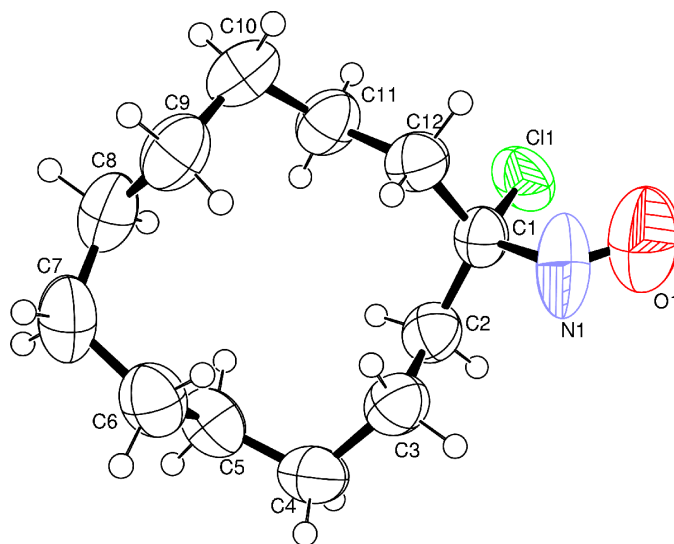
## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
Disorder in main residue  
 $R$  factor = 0.054  
 $wR$  factor = 0.155  
Data-to-parameter ratio = 18.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**gem-Chloronitrosocyclododecane,**  
**(CH<sub>2</sub>)<sub>n-1</sub>CNOCl, with  $n = 12$** The title compound, C<sub>12</sub>H<sub>22</sub>CINO, adopts the square conformation [3333] observed in other known saturated twelve-membered rings. Disorder is observed, resulting from exchange of the chloro and nitroso substituents.

## Comment

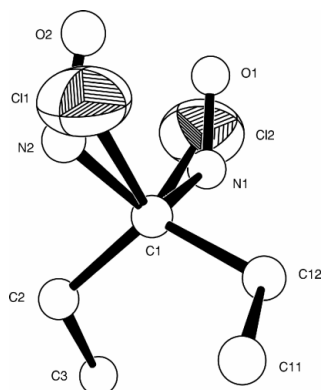
*gem*-Chloronitroso compounds are used in organic syntheses and mechanistic studies, particularly as precursors of aliphatic nitro derivatives (Marchand & Suri, 1984; Marchand *et al.*, 1988) and of vicinal dinitro compounds (Wade *et al.*, 1987; Paquette *et al.*, 1988).

The molecular structure of the title compound, (I), is shown in Fig. 1. The torsion angles (Table 1) indicate that the cyclododecane ring is in a square conformation [3333], as in other saturated twelve-membered rings reported previously (Groth, 1979*a,b*, 1980). Although the skeleton of the cyclododecane ring does not exhibit any observable disorder, the chloro and nitroso groups are disordered (Fig. 2). There are two possible orientations, Cl1/N1/O1 and Cl2/N2/O2, each with 50% occupancy. This can be explained as an orientational

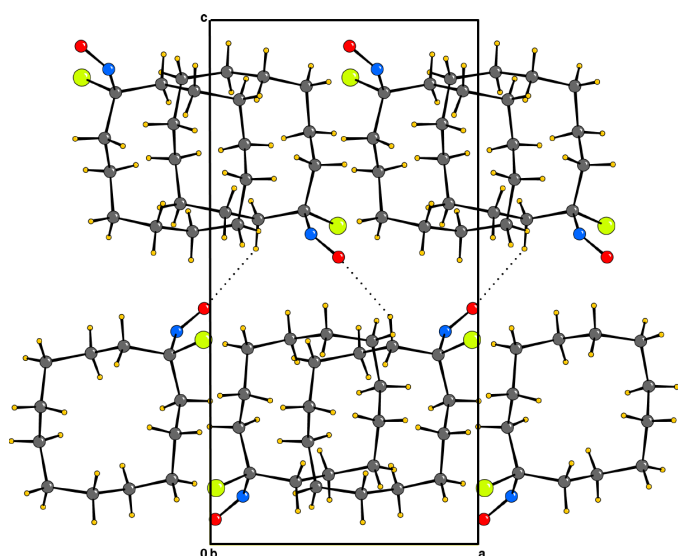


**Figure 1**  
Molecular structure of (I), showing 50% probability displacement ellipsoids. Only one orientation of the disordered chloro and nitroso groups is shown.

Received 9 August 2004  
Accepted 7 September 2004  
Online 18 September 2004



**Figure 2**  
The disorder of the chloro and nitroso groups.



**Figure 3**  
Crystal structure of (I), viewed down the *b* axis. Dotted lines indicate H bonds.

disorder of the molecule around the local pseudo-twofold axis passing through atoms C1 and C7, exchanging the two substituents. Fig. 3 shows the packing of the molecules, which is stabilized by C—H···O interactions (Table 2) and van der Waals forces.

## Experimental

The title compound, (I), was synthesized by a reported procedure (Karim, 2003). In a 100 ml two-necked flask, 10 mmol of cyclododecanoneoxime in 10 ml of dry diethyl ether was cooled to 263 K in an ice-salt bath; chlorotrimethylsilane (12 mmol) was introduced through a dropping funnel. The mixture was stirred magnetically, and isoamyl nitrite (12 mmol) was slowly introduced through a dropping funnel over 15 min. The reaction mixture was left to stir for 1.5 h. Water (10 ml) was added. The organic layer, after separation, was washed with water (20 ml) twice and dried over Na<sub>2</sub>SO<sub>4</sub>. The solvent was removed on a rotary evaporator under reduced pressure, and the residue was chromatographed on silica gel (60–120 mesh). Elution with petroleum ether (b.p. 313–333 K) moved down a blue band, which was identified as (I). Single crystals of (I) were grown from a chloroform solution by slow evaporation.

## Crystal data

C<sub>12</sub>H<sub>22</sub>ClNO  
*M<sub>r</sub>* = 231.76  
 Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>  
*a* = 8.3801 (19) Å  
*b* = 9.543 (2) Å  
*c* = 16.348 (4) Å  
*V* = 1307.4 (5) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.177 Mg m<sup>-3</sup>

Mo *K*α radiation  
 Cell parameters from 600 reflections  
 $\theta$  = 2.5–28.1°  
 $\mu$  = 0.27 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Block, blue  
 0.30 × 0.20 × 0.15 mm

## Data collection

Bruker SMART APEX diffractometer  
 $\varphi$ - $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
*T<sub>min</sub>* = 0.821, *T<sub>max</sub>* = 0.960  
 11 518 measured reflections

3072 independent reflections  
 1998 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.055  
 $\theta_{\max}$  = 28.1°  
*h* = -10 → 10  
*k* = -12 → 11  
*l* = -21 → 21

## Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.054  
*wR* (*F*<sup>2</sup>) = 0.155  
*S* = 0.95  
 3072 reflections  
 163 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0985P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.29 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.25 \text{ e \AA}^{-3}$   
 Absolute structure: Flack (1983),  
 1239 Friedel pairs  
 Flack parameter = 0.00 (10)

**Table 1**

Selected torsion angles (°).

C12—C1—C2—C3	-62.1 (3)	C6—C7—C8—C9	-63.5 (3)
C1—C2—C3—C4	156.64 (19)	C7—C8—C9—C10	158.4 (2)
C2—C3—C4—C5	-74.4 (3)	C8—C9—C10—C11	-72.5 (3)
C3—C4—C5—C6	-73.1 (3)	C9—C10—C11—C12	-74.4 (3)
C4—C5—C6—C7	162.5 (2)	C10—C11—C12—C1	161.1 (2)
C5—C6—C7—C8	-64.3 (3)	C2—C1—C12—C11	-64.1 (3)

**Table 2**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C2—H2B···O1 <sup>i</sup>	0.97	2.69	3.533 (8)	145
C3—H3B···O2 <sup>i</sup>	0.97	2.71	3.467 (8)	135
C4—H4A···O2 <sup>ii</sup>	0.97	2.91	3.511 (8)	121

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

The chloro and nitroso groups show disorder and there are two possible orientations, C11/N1/O1 and C12/N2/O2, each with 50% occupancy. Fitting the light atoms of the nitroso group near the heavier Cl atom yielded large displacement parameters. Restraints were applied for C—N and N—O bond distances to give reasonable values. The N and O atoms were refined anisotropically with restraints for approximate isotropy. H atoms were positioned geometrically and treated as riding, with C—H distances of 0.97 Å and *U<sub>iso</sub>*(H) = 1.2*U<sub>eq</sub>*(parent).

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

CRG thanks UGC-FIP for a Teacher fellowship. The authors thank the Department of Science and Technology, India, for data collection on the CCD facility set up under the IRPHA DST program. They also thank Professor K. Sivakumar, Department of Physics, Anna University, Chennai, for useful suggestions and discussions.

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