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## The Novel Fragrance 2-Isobutyl-4-oxo-5,5-dimethyltetrahydropyran as its 2,4-Dinitrophenylhydrazone Derivative

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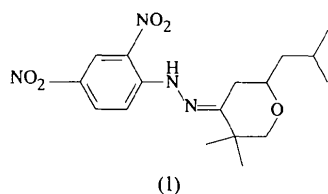
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### Abstract

The structure of the title compound, 2-isobutyl-5,5-dimethyltetrahydropyran-4-one 2,4-dinitrophenylhydrazone, C<sub>17</sub>H<sub>24</sub>N<sub>4</sub>O<sub>5</sub>, was determined and the identity of the 2-isobutyl substituent, unclear from NMR spectroscopy, was confirmed.

### Comment

2-Isobutyl-4-oxo-5,5-dimethyltetrahydropyran was prepared and organoleptically evaluated by Quest International as a potential novel fragrance ingredient. The evaluation was carried out by a panel of expert perfumers. The compound possessed a fruity odour of moderate intensity which, although predominantly rhubarb in character, was also perceived as having grapefruit, chemical, nitrile and urinous notes. The hybridization at C23, and hence the identity of the 2-isobutyl group, could not be assigned reliably from a proton NMR spectrum. In order to confirm the structure by X-ray crystallography, the ketone was converted into the corresponding crystalline 2,4-dinitrophenylhydrazone derivative, (1) (Fig. 1).



All H atoms, including those on C23, were evident in a difference synthesis and the bond lengths for C1—C23 and C23—C24 confirmed *sp*<sup>3</sup> hybridization at C23.

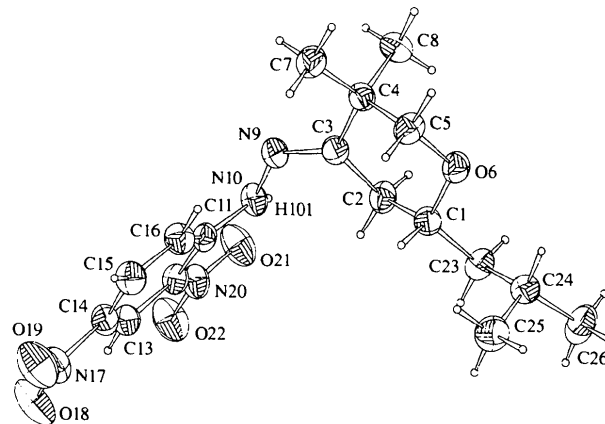


Fig. 1. The molecular structure of (1) with 50% probability displacement ellipsoids.

### Experimental

The potential fragrance was prepared as a liquid by a standard six-step synthesis, starting from isopropyl methyl ketone (Rossiter, 1997) and converted to the corresponding 2,4-dinitrophenylhydrazone derivative. This was crystallized from ethanol/*n*-hexane.

#### Crystal data

C<sub>17</sub>H<sub>24</sub>N<sub>4</sub>O<sub>5</sub>  
*M<sub>r</sub>* = 364.40  
 Triclinic  
*P* $\bar{1}$   
*a* = 6.681 (3) Å  
*b* = 9.093 (1) Å  
*c* = 16.067 (3) Å  
 $\alpha$  = 70.89 (1)°  
 $\beta$  = 75.48 (3)°  
 $\gamma$  = 83.96 (4)°  
*V* = 892.5 (5) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.36 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

#### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction: refined from  $\Delta F$  (Walker & Stuart, 1983)  
*T<sub>min</sub>* = 0.67, *T<sub>max</sub>* = 0.92  
 4804 measured reflections  
 3415 independent reflections

#### Refinement

Refinement on *F*<sup>2</sup>  
*R*(*F*) = 0.055  
 $\omega R$ (*F*<sup>2</sup>) = 0.177

Cu *K*α radiation  
 $\lambda$  = 1.5418 Å  
 Cell parameters from 23 reflections  
 $\theta$  = 15–60°  
 $\mu$  = 0.80 mm<sup>-1</sup>  
*T* = 293 K  
 Plate  
 0.60 × 0.50 × 0.10 mm  
 Red

*R<sub>int</sub>* = 0.019  
 $\theta_{max}$  = 71.93°  
*h* = -7 → 8  
*k* = -10 → 11  
*l* = -11 → 19  
 3 standard reflections  
 frequency: 60 min  
 intensity decay: 6.7%

$(\Delta/\sigma)_{max}$  < 0.001  
 $\Delta\rho_{max}$  = 0.38 e Å<sup>-3</sup>  
 $\Delta\rho_{min}$  = -0.24 e Å<sup>-3</sup>

$S = 0.741$   
 3415 reflections  
 236 parameters  
 H atoms not refined, except  
 H101 on N10  
 Weighting by Chebychev  
 polynomial (Carruthers &  
 Watkin, 1979)

Extinction correction:  
 Larson (1970)  
 Extinction coefficient:  
 174 (14)  
 Scattering factors from *Inter-*  
*national Tables for X-ray*  
*Crystallography* (Vol. IV)

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## A *trans*-1,2-Diarylhexafluorocyclobutane

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Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

C1—C23	1.517 (2)	C23—C24	1.529 (2)
C1—C23—C24	114.5 (1)		

All H atoms were located in a difference map, although those connected to C were replaced at ideal positions. A local implementation of the *DIFABS* algorithm (Walker & Stuart, 1983), which evaluates a contribution to  $F_c$  (rather than  $F_o$ ), was applied to the unmerged data and the structure refined to convergence with a Chebychev weighting scheme (Carruthers & Watkin, 1979). At convergence, the reciprocal absorption correction was applied to  $F_o$ , equivalent reflections merged and the final difference synthesis computed.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *RC93* (Watkin, Prout & Lilley, 1994). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *CRYSTALS* (Watkin, Prout, Carruthers & Betteridge, 1996). Molecular graphics: *CAMERON* (Watkin, Prout & Pearce, 1996). Software used to prepare material for publication: *CRYSTALS*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1165). Services for accessing these data are described at the back of the journal.

## References

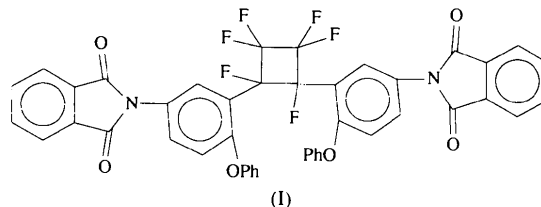
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## Abstract

Crystal structure analysis establishes the configuration of one of two isomers of the thermal dimerization of *N*-[3-(1,2,2-trifluoroethyl)-4-phenoxyphenyl]phthalimide to be *trans*-5,5'-[1,2,3,3,4,4-hexafluorocyclobutane-1,2-diylbis(2-phenoxyphenyl)]-*N,N'*-bis(phthalimide),  $C_{44}H_{24}F_6N_2O_6$ . The cyclobutane ring puckers and has a dihedral angle of  $23.5(4)^\circ$ . The crystal also contains a diethyl ether molecule of solvation,  $C_4H_{10}O$ .

## Comment

*N*-[3-(1,2,2-Trifluoroethyl)-4-phenoxyphenyl]phthalimide undergoes [2+2] cycloaddition to give a major and a minor product (64 and 5% yield, respectively) that are *cis/trans* isomers of the octa-substituted cyclobutane. Recrystallization of the major product from ether gave crystals suitable for X-ray diffraction analysis. On consideration of steric interactions between adjacent aryl substituents, one would predict that the *trans* isomer would be the major product. This prediction was confirmed as the crystal structure determination established the major isomer to be *trans*-5,5'-[1,2,3,3,4,4-hexafluorocyclobutane-1,2-diylbis(2-phenoxyphenyl)]-*N,N'*-bis(phthalimide), (I). A diethyl ether molecule of solvation is also included in the crystal.



The cyclobutane ring is puckered. The C1—C2—C3 plane forms a dihedral angle of  $23.5(4)^\circ$  with the C3—C4—C1 plane. A survey of the geometry of cyclobutanes (Allen, 1984) reports the average pucker in acyclic-substituted cyclobutane rings to be  $24.3^\circ$ . The aryl substituents have three planar regions. For the aryl group bonded to the C1 cyclobutane atom, the plane of the phenoxyphenyl group forms a dihedral angle of  $67.3(1)^\circ$  with the plane of the phenyl ring bonded to the