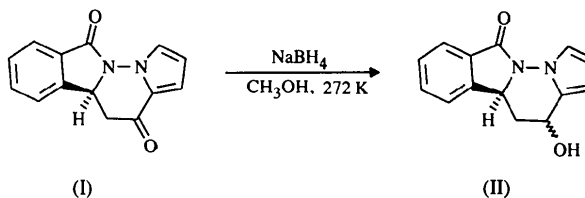


Sheldrick, G. M. (1994). *SHELXTL. Structure Determination Programs*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1995). *ASTRO and SAINT. Data Collection and Processing Software for the SMART System*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Webster, M. S. (1956). *J. Chem. Soc.* pp. 2841–2845.

its structure was supported by <sup>1</sup>H and <sup>13</sup>C NMR spectral data. To confirm the *cis* configuration of the hydroxy group and the isoindole ring, the X-ray diffraction study of (II) was undertaken.



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### *cis*-6,10b,11,12-Tetrahydro-12-hydroxy-pyrrolo[1',2':2,3]pyridazino[6,1-*a*]isoindol-6-one

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#### Abstract

The title molecule, C<sub>14</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>, consists of a pyridazine ring fused to a five-membered pyrrole ring and to an isoindole moiety. The pyrrole ring and the isoindole moiety are planar, defining a dihedral angle of 35°, while the pyridazine ring adopts an envelope conformation. The isoindole ring and the hydroxy group are in a *cis* configuration.

#### Comment

It has been reported that isoindolo[2,1-*a*]quinoline derivatives similar to vinpocetine (Karpati & Szporny, 1976) have vasodilator activity (Ishihara, Kiyota & Goto, 1990). These results, in connection with our studies on polycyclic heterocycles containing a pyrrole ring, prompted us to search for pyrrolo analogues of this system. The stereoselective reduction of racemic 10b,11-dihydropyrrolo[1',2':2,3]pyridazino[6,1-*a*]isoindole-6(6*H*),12-dione, (I), with NaBH<sub>4</sub> in methanol at 272 K led to the title compound, (II) (Marchalín & Decroix, 1995). Only one diastereomer was isolated and

The bond lengths are consistent with average values in the usual sources [*International Tables for Crystallography* (1992, Vol. C, Table 9.5.1.1)]. Selected torsion angles are given in Table 1.

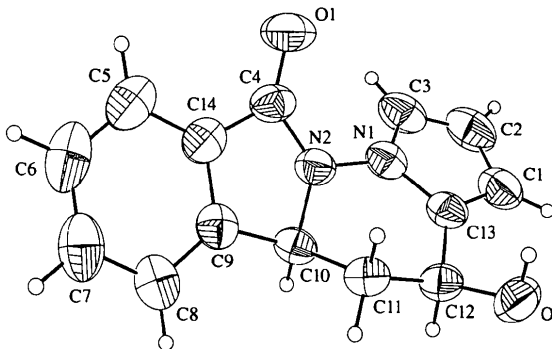


Fig. 1. The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

#### Experimental

The title compound was prepared by stereoselective reduction of racemic 10b,11-dihydropyrrolo[1',2':2,3]pyridazino[6,1-*a*]isoindole-6(6*H*),12-dione, (I), with NaBH<sub>4</sub> in methanol at 272 K.

#### Crystal data

C<sub>14</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>  
M<sub>r</sub> = 240.26  
Monoclinic  
P2<sub>1</sub>/n  
a = 8.386 (2) Å  
b = 8.149 (2) Å  
c = 17.267 (4) Å  
β = 99.04 (4)°  
V = 1165.3 (5) Å<sup>3</sup>  
Z = 4  
D<sub>x</sub> = 1.369 Mg m<sup>-3</sup>  
D<sub>m</sub> not measured

Mo Kα radiation  
λ = 0.71069 Å  
Cell parameters from 25 reflections  
θ = 17.3–21.6°  
μ = 0.094 mm<sup>-1</sup>  
T = 298 (2) K  
Block  
1.32 × 0.80 × 0.57 mm  
Colourless

**Data collection**

Stoe Stadi-4 diffractometer	$\theta_{\max} = 23.05^\circ$
$\omega/2\theta$ scans	$h = -9 \rightarrow 9$
Absorption correction: none	$k = -6 \rightarrow 8$
2573 measured reflections	$l = -14 \rightarrow 19$
1603 independent reflections	3 standard reflections
1454 reflections with $I > 2\sigma(I)$	frequency: 120 min
$R_{\text{int}} = 0.012$	intensity decay: none

**Refinement**

Refinement on $F^2$	$(\Delta/\sigma)_{\max} = 0.054$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$\Delta\rho_{\max} = 0.11 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.090$	$\Delta\rho_{\min} = -0.12 \text{ e } \text{\AA}^{-3}$
$S = 1.045$	Extinction correction:
1601 reflections	<i>SHELXL93</i> (Sheldrick, 1993)
168 parameters	Extinction coefficient:
H-atom parameters refined only for H21	0.039 (3)
$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.3151P]$	Scattering factors from
where $P = (F_o^2 + 2F_c^2)/3$	<i>International Tables for Crystallography</i> (Vol. C)

Table 1. Selected torsion angles ( $^\circ$ )

C13—N1—N2—C10	-20.3 (2)	C10—C11—C12—O2	171.38 (12)
C4—N2—C10—C11	-107.46 (14)	C10—C11—C12—C13	44.4 (2)
N1—N2—C10—C11	48.8 (2)	C11—C12—C13—C1	173.3 (2)
N2—C10—C11—C12	-61.0 (2)	C11—C12—C13—N1	-15.2 (2)
C9—C10—C11—C12	-172.55 (12)		

Data collection: *Enraf-Nonius SDP-Plus Structure Determination Package* (Frenz, 1985). Cell refinement: *SHELXL93* (Sheldrick, 1993). Data reduction: *REDU4* (Stoe & Cie, 1988). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93*. Molecular graphics: *ORTEPII* (Johnson, 1971). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1289). Services for accessing these data are described at the back of the journal.

**References**

- Frenz, B. A. (1985). *Enraf-Nonius SDP-Plus Structure Determination Package*. Version 3.0. Enraf-Nonius, Delft, The Netherlands.
- Ishihara, Y., Kiyota, Y. & Goto, G. (1990). *Chem. Pharm. Bull.* **38**, 3024–3030.
- Johnson, C. K. (1971). *ORTEPII*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee, USA.
- Karpati, E. & Szpomy, L. (1976). *Arzneim. Forsch.* **26**, 1908–1912.
- Marchalín, S. & Decroix, B. (1995). *Heterocycles*, **41**, 689–696.
- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Stoe & Cie (1988). *REDU4. Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

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## 1,3,3-Trimethyl-5-styryl-2-[(1,3,3-trimethyl-5-styryl-2-indolinylidene)methyl]-3H-indolium Picrate: an Extended Trichromophoric System

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**Abstract**

The trichromophoric system of the title compound,  $C_{39}H_{39}N_7^+ \cdot C_6H_2N_3O_7^-$ , adopts a di-*cis* conformation in which the indolenine end groups are mutually rotated; the interplanar angle is  $47.1(1)^\circ$ . The *trans*-configurate styryl moieties are *anti* orientated relative to the cyanine unit. The length of the extended chromophore (H19···H19') is 26.0 Å.

**Comment**

Organic non-linear materials possess promising properties for a wide range of applications. Cyanines and other polymethine dyes exhibit interesting third-order non-linear properties (Nalwa, 1993; Dirk *et al.*, 1995; Kawabe, Sakai, Ikeda, Hasegawa & Kawasaki, 1993; Werncke, Pfeiffer, Lau, Grahn & Johannes, 1997). In order to investigate structure–property relationships, we have developed new methods of functionalizing cyanines while conserving the chromophore (Grahn, Johannes, Rheinheimer, Knieriem & Würthwein, 1995; Johannes, Grahn, Reisner & Jones, 1995). Since polymethines that are halogenated in the chains and/or at the end groups are easily accessible (Tyutyulkov, Fabian, Mehlhorn, Dietz & Tadjer, 1991; Grahn *et al.*, 1995), we can introduce carbon substituents into the chain and/or the end groups by palladium-catalyzed cross-coupling reactions (Johannes & Grahn, 1997).

The title compound, (1a), was prepared by Heck reaction of the 5,5'-diiodoindocyanine (2) with an excess of styrene in DMF. The tetrafluoroborate counterion was replaced by picrate in order to fix the anion to the cyanine cation by  $\pi$ -stacking (In, Nagata, Doi, Ishida & Wakahara, 1997). We report here the crystal structure of (1a).

The cyanine adopts a di-*cis* conformation in the solid state as in solution (Johannes & Grahn, 1997); the