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6-Diethylamino-1-dimethylthiocarbamoyl-5-methyl-3-phenyl-1*H*-pyridazin-4-one (I), $C_{18}H_{24}N_4OS$, and 5-Diethylthiocarbamoyl-6-dimethylamino-5-methyl-3-phenyl-5*H*-pyridazin-4-one (II), $C_{18}H_{24}N_4OS$*

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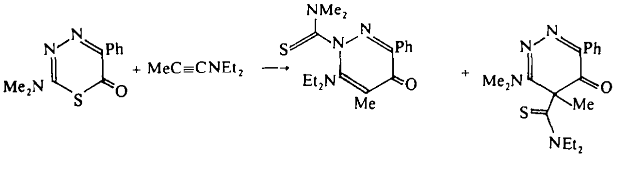
(*Received* 13 *February* 1985; *accepted* 10 *May* 1985)

Abstract. (I): $M_r = 344{\cdot}5$, monoclinic, $P2_1/c$, $a = 13{\cdot}210\,(1)$, $b = 10{\cdot}353\,(1)$, $c = 14{\cdot}386\,(1)$ Å, $\beta = 110{\cdot}586\,(3)°$, $V = 1841{\cdot}8\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}242$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 1{\cdot}61$ mm^{-1}, $F(000) = 736$, $T = 293\,(1)$ K, final $R = 0{\cdot}069$ for 2432 significant reflections. (II): $M_r = 344{\cdot}5$, monoclinic, $P2_1/n$, $a = 11{\cdot}154\,(1)$, $b = 14{\cdot}943\,(1)$, $c = 11{\cdot}066\,(1)$ Å, $\beta = 102{\cdot}239\,(3)°$, $V = 1802{\cdot}5\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}269$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1{\cdot}54178$ Å, $\mu = 1{\cdot}65$ mm^{-1}, $F(000) = 736$, $T = 293\,(1)$ K, final $R = 0{\cdot}054$ for 2446 significant reflections. Both compounds are formed by the reaction of 2-dimethylamino-5-phenyl-1,3,4-thiadiazin-6-one with 1-(diethylamino)propyne. Whereas in (I) the six-membered heterocyclic ring is planar, in (II), with a tetrahedral C atom at the 5-position, there is considerable ring puckering and incomplete conjugation over the remaining ring atoms is quite pronounced.

Introduction. We recently reported (Baydar, Boyd & Lindley, 1981) that the novel 1,3,4-thiadiazinone (III) reacted with 1-(diethylamino)propyne to yield a mixture of the rearranged adducts (I) and (II). Compound (II) is particularly interesting, as it is not fully conjugated; such 5*H*-pyridazin-4-ones have not been described previously. We now give details of the X-ray analyses and molecular structures of the two isomeric adducts.



(III) (I) (II)

Experimental. X-ray data collection and refinement details are listed in Table 1. Details of the preparation of compounds (I) and (II) and spectroscopic data have

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* IUPAC names: (I) 6-Diethylamino-*N*,*N*,5-trimethyl-4-oxo-3-phenyl-1,4-dihydro-1-pyridazinecarbothioamide. (II) 3-Dimethylamino-*N*,*N*-diethyl-4-methyl-5-oxo-6-phenyl-4,5-dihydro-4-pyridazinecarbothioamide.

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Table 1. *Data collection and refinement details for compounds* (I) *and* (II)

	(I)	(II)
(*a*) Data collection		
Max. cross-sectional diameter of crystal fragment (mm)	0·5	0·5
Scan step size (°) and moving-window parameters	0·02; 54 (36)	0·02; 60 (40)
Number and θ range (°) of lattice-parameter reflections	20; 28·0–35·0	20; 30·1–34·6
Absorption correction: max. % variation in transmission	26·0	33·8
Max. sin θ/λ (Å^{-1})	0·59	0·59
Ranges of h	−15; 15	−10; 13
k	−9; 11	−13; 17
l	0; 16	−12; 10
% variation of intensity sums of four reference reflections	17·5	11·1
Total number of reflections measured	4828	4990
Number of unique *hkl*	3047	3040
Merging R for equivalent *hkl*	0·029	0·021
Number of unobserved *hkl*, $I < 3\sigma(I)$	615	594
(*b*) Refinement		
Number of significant *hkl*	2432	2446
R	0·069	0·054
wR	0·088	0·063
S	0·45	0·29
Extinction parameter, g ($\times 10^5$)	3·2 (4)	2·9 (3)
Weighting coefficients, a	20·0	20·0
b	50·0	50·0
c	0·002	0·002
$F_{\min}$	0	20
d	0	0·004
Max. shift/error in final least-squares cycle	0·74	0·22
Max. and min. electron density in final difference Fourier synthesis (e Å^{-3})	0·3; −0·3	0·2; −0·2

been deposited.* Compounds (I) and (II) obtained as large crystalline aggregates, approximately equidimensional fragments used for the X-ray studies; preliminary crystal data from Weissenberg and precession photographs. Hilger & Watts Y290 diffractometer, Cu $K\alpha$ nickel-filtered radiation, $\omega/2\theta$ step scans, count time 1 s per step, moving-window estimation of intensity and background (Tickle, 1975). Reference reflections measured every 50 reflections and data scaled to the reference intensity sums by interpolation. Lp and empirical absorption corrections (North, Phillips & Mathews, 1968) for all data. Structures solved by direct methods with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinement (modified version of program by D. W. J. Cruickshank and J. G. F. Smith) using only significant reflections. H atoms located by difference Fourier syntheses but constrained to lie in geometricized positions assuming C—H 1·0 Å. All non-H atoms refined anisotropically, $\sum w(|F_o|-|F_c|)^2$ minimized, $w=d$ if $|F_o|<F_{\min}$ else $w=[1-\exp(-a\sin^2\theta/\lambda^2)]/(b+|F_o|+c|F_o|^2)$ (see Table 1). Empirical extinction corrections, g, applied by multiplying uncorrected structure factors, F_c', by $(1+g|F_c'|)^{-1/2}$. Scattering factors from Hanson, Herman, Lea & Skillman (1964); all computations carried out on the CDC 6600 computer at the University of London Computer Centre.

Discussion. Atomic positional parameters and equivalent isotropic thermal parameters for (I) and (II) are listed in Table 2.* Figs. 1(*a*) and 1(*b*) show the atomic labelling for (I) and (II) and the bond lengths and angles. Figs. 2(*a*) and 2(*b*) are stereoscopic drawings of the molecules of (I) and (II) respectively and clearly show the planarity of the pyridazine ring in (I) compared to the pronounced puckering in (II). In (II) the pyridazine ring adopts a distorted sofa-like conformation in which the dominant symmetry element is a pseudo-mirror plane through N(1) and C(4); endo-

Table 2. *Fractional coordinates and equivalent isotropic thermal parameters* (Å$^2\times 10^3$) *for the non*-H *atoms with e.s.d.'s in parentheses*

$$U_{\rm eq}=\tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
(*a*) Compound (I)				
N(1)	0·7630 (2)	−0·1932 (3)	0·4172 (2)	39 (1)
C(11)	0·7410 (3)	−0·0555 (3)	0·4101 (2)	40 (2)
S(1)	0·6392 (1)	0·0012 (1)	0·3168 (1)	61 (1)
N(12)	0·8137 (3)	0·0141 (3)	0·4796 (2)	50 (2)
C(13)	0·8972 (4)	−0·0380 (4)	0·5677 (3)	69 (2)
C(14)	0·8098 (5)	0·1551 (4)	0·4730 (4)	72 (2)
N(2)	0·8524 (2)	−0·2209 (3)	0·3951 (2)	40 (2)
C(3)	0·8678 (3)	−0·3390 (3)	0·3730 (2)	41 (2)
C(31)	0·9652 (3)	−0·3601 (3)	0·3459 (2)	41 (2)
C(32)	1·0301 (3)	−0·4684 (4)	0·3780 (3)	50 (2)
C(33)	1·1234 (3)	−0·4814 (4)	0·3539 (3)	60 (2)
C(34)	1·1502 (3)	−0·3897 (5)	0·2980 (3)	61 (2)
C(35)	1·0861 (4)	−0·2821 (5)	0·2657 (3)	57 (2)
C(36)	0·9943 (3)	−0·2670 (4)	0·2905 (3)	49 (2)
C(4)	0·7936 (3)	−0·4453 (3)	0·3716 (3)	46 (2)
O(41)	0·8094 (3)	−0·5565 (3)	0·3486 (3)	70 (2)
C(5)	0·7045 (3)	−0·4112 (3)	0·4021 (3)	45 (2)
C(51)	0·6356 (4)	−0·5208 (4)	0·4150 (4)	59 (2)
C(6)	0·6893 (3)	−0·2858 (3)	0·4229 (2)	40 (2)
N(61)	0·6056 (2)	−0·2410 (3)	0·4515 (2)	45 (2)
C(62)	0·6278 (5)	−0·1643 (6)	0·5413 (4)	78 (3)
C(63)	0·6079 (9)	−0·2273 (9)	0·6217 (5)	132 (5)
C(64)	0·4947 (4)	−0·2727 (5)	0·3866 (4)	77 (3)
C(65)	0·4165 (4)	−0·1660 (6)	0·3730 (5)	94 (3)
(*b*) Compound (II)				
N(1)	0·1299 (2)	−0·0772 (2)	0·9756 (2)	41 (1)
N(2)	0·1587 (2)	−0·0023 (2)	1·0468 (2)	42 (1)
C(3)	0·2004 (3)	0·0717 (2)	1·0095 (2)	35 (1)
C(31)	0·2269 (2)	0·1467 (2)	1·0988 (3)	37 (1)
C(32)	0·2368 (3)	0·1306 (2)	1·2244 (3)	45 (1)
C(33)	0·2621 (3)	0·1998 (3)	1·3087 (3)	55 (1)
C(34)	0·2781 (3)	0·2860 (3)	1·2709 (3)	57 (1)
C(35)	0·2658 (3)	0·3034 (2)	1·1458 (3)	51 (1)
C(36)	0·2404 (3)	0·2344 (2)	1·0609 (3)	43 (1)
C(4)	0·2300 (3)	0·0770 (2)	0·8866 (3)	37 (1)
O(41)	0·2995 (3)	0·1311 (2)	0·8577 (2)	62 (1)
C(5)	0·1639 (2)	0·0086 (2)	0·7913 (2)	33 (1)
C(51)	0·2491 (2)	−0·0060 (2)	0·6994 (2)	34 (1)
S(1)	0·2129 (1)	0·0368 (1)	0·5575 (1)	49 (1)
N(52)	0·3534 (2)	−0·0509 (2)	0·7420 (2)	37 (1)
C(53)	0·3993 (3)	−0·0830 (2)	0·8693 (3)	50 (1)
C(54)	0·4067 (4)	−0·1840 (3)	0·8798 (4)	74 (2)
C(55)	0·4446 (3)	−0·0608 (2)	0·6634 (3)	48 (2)
C(56)	0·5254 (3)	0·0210 (3)	0·6703 (4)	64 (2)
C(57)	0·0410 (3)	0·0562 (2)	0·7351 (3)	44 (1)
C(6)	0·1299 (2)	−0·0749 (2)	0·8556 (3)	34 (1)
N(61)	0·0946 (2)	−0·1497 (2)	0·7924 (2)	39 (1)
C(62)	0·0667 (3)	−0·2301 (2)	0·8565 (3)	51 (1)
C(63)	0·0701 (3)	−0·1595 (2)	0·6580 (3)	50 (1)

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and preparative details have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42236 (37 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* See deposition footnote.

cyclic torsion angles are given in Table 3. C(4) and C(5) lie 0·276 (6) and −0·089 (5) Å respectively out of the least-squares plane through atoms N(1), N(2), C(3) and C(6). The endocyclic bond lengths indicate incomplete conjugation over the ring atoms [excluding C(5)]; the N(2)–C(3) separation, 1·300 (4) Å, is very close to the value of 1·29 Å expected for a C=N double bond and the C(3)–C(4) and C(4)–O(41) distances, 1·468 (4) and 1·209 (4) Å respectively, are typical of C–C single and C=O double bonds involving sp^2-hybridized C atoms. The environment about C(6), however, is almost planar and the N(1)–C(6) and C(6)–N(61) distances, 1·328 (4) and 1·333 (4) Å respectively, indicate considerable electron delocalization over this part of the molecule.

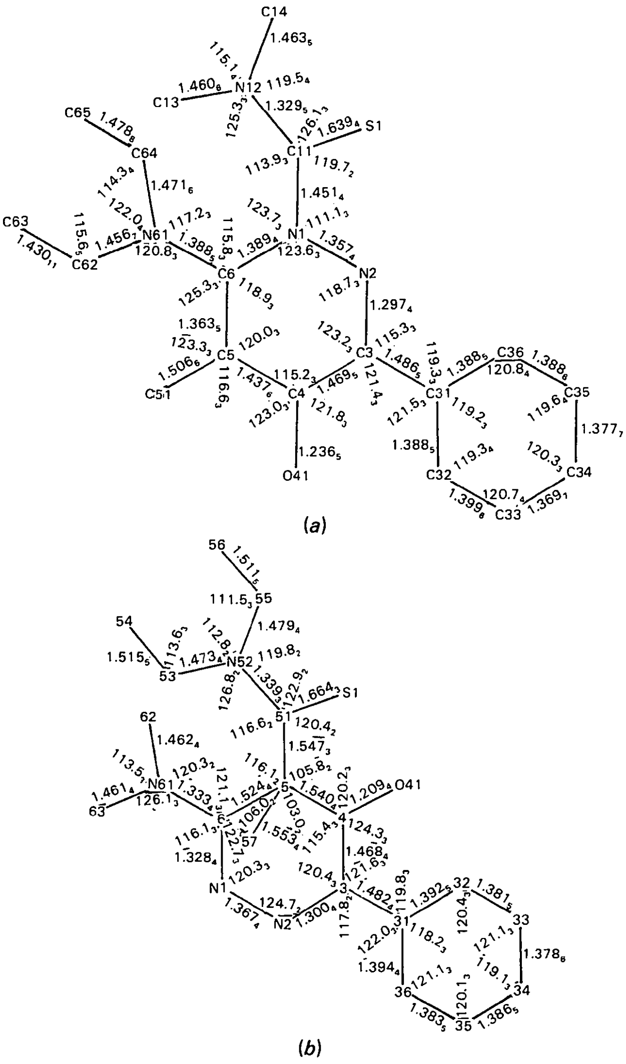

Fig. 1. Schematic drawings of the molecules of (*a*) compound (I), and (*b*) compound (II). Bond distances in Å, angles in °. The angles not shown in (*b*) are C(4)–C(5)–C(6) = 110·7 (2) and C(51)–C(5)–C(57) = 114·5 (2)°. E.s.d.'s are shown as subscripts.

In (I) the heterocyclic ring is minimally puckered (see Table 3), the maximum displacement out of the least-squares ring plane being 0·032 (5) Å for C(4), but in a similar manner to (II) the N(2)–C(3) linkage, 1·297 (4) Å, is typical of a C=N bond. The C(5)–C(6) bond, 1·363 (5) Å, also exhibits considerable double-bond character but the bonds adjacent to these two atoms and the C(4)–O(41) separation [0·027 (4) Å longer than in (II)] suggest appreciable electron delocalization.

The geometry of the thiocarbamoyl moiety is closely similar in (I) and (II) and the bond distances within the S–C–N group – average S–C and C–N distances are 1·652 (5) and 1·334 (5) Å respectively – together with the planarity at the central C atom indicate electron delocalization over these three atomic centres. In (I) the dihedral angle between the plane of the thiocarbamoyl group (defined by the central C atom and its three nearest neighbours) and the pyridazine ring is 67·8 (1)°, whereas in (II) it makes a dihedral angle of 79·9 (1)° with the least-squares plane through N(1), N(2), C(3) and C(6). The corresponding dihedral

Table 3. *Endocyclic torsion angles* (°) *for the pyridazine rings in compounds* (I) *and* (II)

	(I)	(II)
N(1)–N(2)–C(3)–C(4)	−0·6 (5)	−5·3 (5)
N(2)–C(3)–C(4)–C(5)	−3·4 (5)	23·2 (4)
C(3)–C(4)–C(5)–C(6)	4·6 (5)	−26·4 (3)
C(4)–C(5)–C(6)–N(1)	−2·1 (5)	15·6 (4)
C(5)–C(6)–N(1)–N(2)	−2·3 (5)	1·4 (4)
C(6)–N(1)–N(2)–C(3)	3·7 (5)	−7·9 (4)

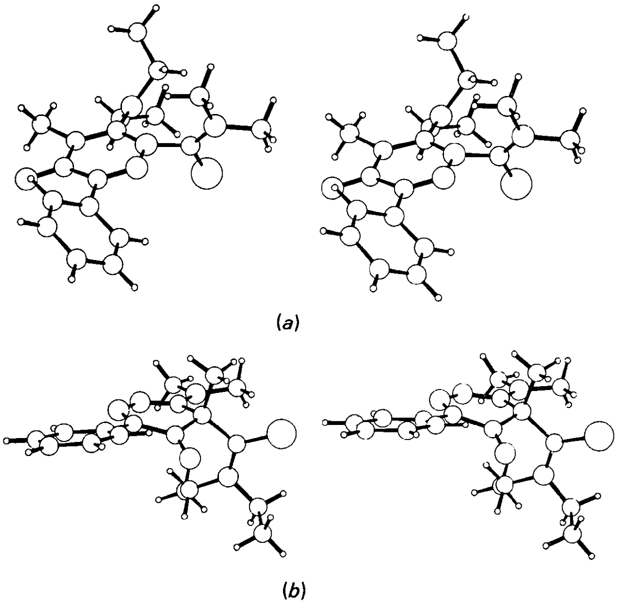

Fig. 2. Stereoscopic drawings of the molecules of (*a*) compound (I), and (*b*) compound (II).

angles for the phenyl groups are 38·9 (1) and 12·6 (2)° respectively.

We thank the SERC for financial support.

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SHORT COMMUNICATIONS

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Acta Cryst. (1985). C**41**, 1280

The structure of ethyl 5,6,7,8,9-pentaacetoxy-2-amino-4-nitromethyl-D-*glycero*-D-*manno*-2-nonene-3-carboxylate, $C_{23}H_{34}N_2O_{14}$: erratum.

By R. VEGA, M. J. DIÁNEZ, A. LÓPEZ-CASTRO and R. MÁRQUEZ, *Departamento de Optica y Sección de Física del Departamento de Investigaciones Físicas y Químicas del CSIC, Universidad de Sevilla, Spain*

(*Received* 11 *March* 1985; *accepted* 29 *April* 1985)

Abstract

An error in the name of the title compound of the paper by Vega, Diánez, López-Castro & Márquez [*Acta Cryst.* (1984), C**40**, 1941–1944] is corrected. The correct name is ethyl (*Z*)-5,6,7,8,9-pentaacetoxy-2-amino-4-nitromethyl-D-*glycero*-D-*ido*-2-nonene-3-carboxylate. Thus, throughout the text D-*glycero*-D-*manno* should read D-*glycero*-D-*ido*

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All relevant information is given in the *Abstract*.

International Union of Crystallography

Acta Cryst. (1985). C**41**, 1280

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