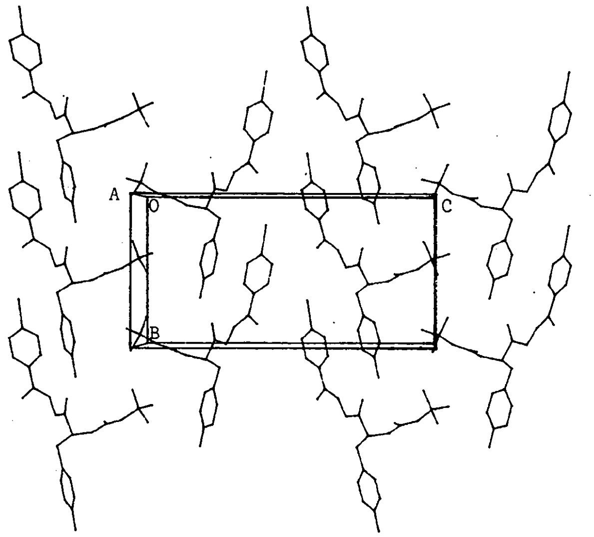

Fig. 2. A view of the unit-cell packing.

crystal packing arrangement are shown in Figs. 1 and 2.

Related literature. For the preparation and properties of the title compound and related derivatives see Nagasawa, Kuroiwa, Narita & Isowa (1973); Stelakatos, Panagou & Zervas (1966); Matsoukas, Moharir & Findlay (1983); Findlay, Dalzeet, Matsoukas & Moharir (1984); Vlassi, Germain, Matsoukas, Psachoulia, Voliotis & Leban (1987); Matsoukas, Goghari, Scanlon, Franklin & Moore (1985); Moore & Matsoukas (1985). Knowledge of the conformation of the title compound and related tyrosine derivatives is desirable for the design and synthesis of effective inhibitors for diagnostic and therapeutic applications.

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1-Mesyl-4-[2-(methoxycarbonyl)ethyl]-3-(methoxycarbonyl)methyl-*N*,*N*-dimethylpyrrole-2-carboxamide

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Abstract. Methyl 3-[5-(*N*,*N*-dimethylamino)carbonyl-1-mesyl-4-(methoxycarbonyl)methyl-3-pyrrolyl]propionate, $C_{15}H_{22}N_2O_7S$, $M_r = 374\cdot42$, orthorhombic, *Pcab* (non-standard setting of *Pbca*, No. 61), $a = 14\cdot015\,(3)$, $b = 14\cdot259\,(3)$, $c = 18\cdot444\,(4)$ Å, $V = 3685\cdot6\,(5)$ Å^3, $Z = 8$, $D_x = 1\cdot35$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha) = 1\cdot54178$ Å, $\mu = 1\cdot864$ mm^{-1}, $F(000) = 1584$, $T = 293$ K. Final $R = 0\cdot073$ for 2250 unique observed $[F > 4\sigma(F)]$ reflexions and 229 parameters. The pyrrole ring is planar [maximum torsion angle = $1\cdot8\,(4)^\circ$]. The carboxamide group is twisted out of the ring plane by $67\cdot5\,(6)^\circ$. The *N*-mesyl unit is rare and this is the first crystallographic study of an *N*-mesylpyrrole. The N—S bond is long at $1\cdot668\,(3)$ Å and corresponds to a shortened mean S=O distance of $1\cdot418\,(3)$ Å as expected from the study of Kálmán, Párkányi & Schawartz [*Acta Cryst.* (1977), B**33**, 3097–3102]. The S(mesyl) tetrahedron is normal [O=S=O = $119\cdot5\,(3)^\circ$, N—S—C = $104\cdot6\,(2)^\circ$, N—S=O(mean) = $106\cdot4\,(3)^\circ$, C—S=O(mean) = $109\cdot5\,(3)^\circ$] and adopts a pseudo-

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Table 1. *Atomic coordinates* ($\times 10^4$, $\times 10^5$ *for* S) *and isotropic thermal parameters* ($Å^2 \times 10^3$)

	x	y	z	U_{eq}*
S(1)	8455 (7)	1975 (6)	63514 (4)	60 (1)
O(1)	423 (2)	−152 (2)	7000 (1)	80 (1)
O(2)	1640 (2)	803 (2)	6397 (2)	89 (1)
C(5)	−29 (3)	714 (3)	5809 (2)	69 (1)
N(1)	1217 (2)	−728 (2)	5876 (2)	60 (1)
C(1)	2061 (3)	−761 (3)	5484 (2)	75 (1)
C(2)	2192 (4)	−1638 (4)	5236 (2)	89 (2)
C(3)	1424 (4)	−2191 (3)	5476 (2)	89 (2)
C(4)	816 (3)	−1631 (2)	5888 (2)	67 (1)
C(6)	−141 (4)	−1806 (3)	6181 (2)	71 (1)
N(2)	−221 (4)	−2404 (3)	6731 (2)	106 (2)
C(14)	2819 (3)	−932 (4)	3727 (3)	96 (2)
O(3)	−831 (2)	−1448 (2)	5886 (2)	86 (1)
C(7)	586 (5)	−2689 (5)	7182 (3)	142 (3)
C(8)	−1185 (5)	−2638 (4)	7002 (4)	132 (3)
C(9)	1250 (6)	−3220 (3)	5340 (3)	136 (3)
C(10)	889 (4)	−3433 (3)	4574 (3)	87 (2)
O(4)	582 (4)	−2891 (2)	4165 (2)	124 (2)
O(5)	940 (3)	−4340 (2)	4453 (2)	100 (1)
C(11)	627 (4)	−4647 (4)	3734 (4)	120 (3)
C(12)	3041 (5)	−1975 (6)	4802 (4)	160 (4)
O(8)	2252 (3)	−858 (4)	3195 (2)	146 (2)
O(7)	3211 (4)	−255 (3)	3946 (4)	161 (2)
C(15)	2186 (6)	110 (6)	2943 (5)	170 (4)
C(13)	2957 (5)	−1902 (4)	4015 (4)	130 (3)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

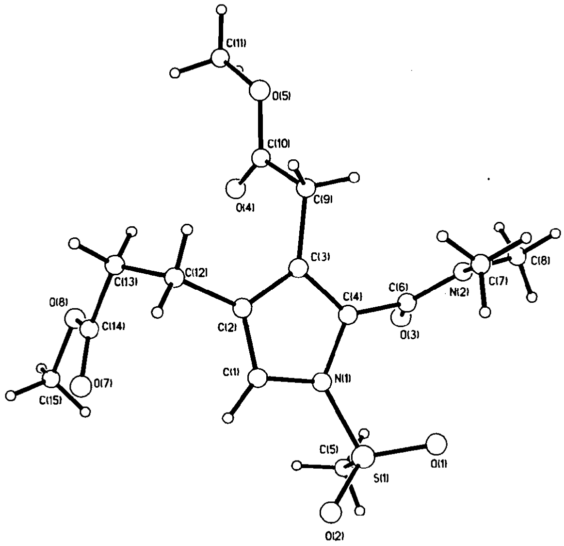


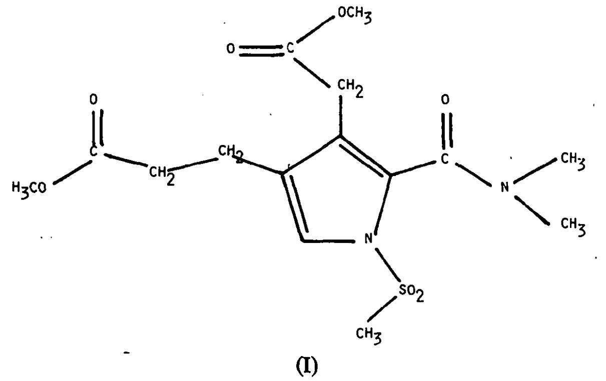
Fig. 1. A perspective view of the molecule showing atomic labelling.

staggered orientation with respect to the pyrrole ring in which the S—methyl vector is almost perpendicular to the ring plane.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

S(1)—O(1)	1·424 (3)	S(1)—O(2)	1·412 (3)
S(1)—C(5)	1·744 (4)	S(1)—N(1)	1·668 (3)
N(1)—C(1)	1·387 (5)	N(1)—C(4)	1·405 (4)
C(1)—C(2)	1·344 (7)	C(2)—C(3)	1·405 (7)
C(2)—C(12)	1·513 (9)	C(3)—C(4)	1·394 (6)
C(3)—C(9)	1·509 (6)	C(4)—C(6)	1·467 (6)
C(6)—N(2)	1·330 (5)	C(6)—O(3)	1·222 (6)
N(2)—C(7)	1·461 (8)	N(2)—C(8)	1·478 (9)
C(14)—O(8)	1·267 (6)	C(14)—O(7)	1·181 (8)
C(14)—C(13)	1·495 (9)	C(9)—C(10)	1·532 (8)
C(10)—O(4)	1·163 (6)	C(10)—O(5)	1·314 (5)
O(5)—C(11)	1·463 (8)	C(12)—C(13)	1·460 (10)
O(8)—C(15)	1·460 (10)		
O(1)—S(1)—O(2)	119·5 (2)	O(1)—S(1)—C(5)	109·7 (2)
O(2)—S(1)—C(5)	109·3 (2)	O(1)—S(1)—N(1)	107·1 (2)
O(2)—S(1)—N(1)	105·6 (2)	C(5)—S(1)—N(1)	104·6 (2)
S(1)—N(1)—C(1)	124·5 (3)	S(1)—N(1)—C(4)	126·3 (3)
C(1)—N(1)—C(4)	108·6 (3)	N(1)—C(1)—C(2)	109·0 (4)
C(1)—C(2)—C(3)	108·1 (4)	C(1)—C(2)—C(12)	125·7 (5)
C(3)—C(2)—C(12)	126·2 (5)	C(2)—C(3)—C(4)	108·6 (4)
C(2)—C(3)—C(9)	128·1 (5)	C(4)—C(3)—C(9)	123·3 (5)
N(1)—C(4)—C(3)	105·8 (4)	N(1)—C(4)—C(6)	121·9 (3)
C(3)—C(4)—C(6)	131·5 (4)	C(4)—C(6)—N(2)	117·9 (4)
C(4)—C(6)—O(3)	119·2 (3)	N(2)—C(6)—O(3)	122·7 (5)
C(6)—N(2)—C(7)	123·1 (5)	C(6)—N(2)—C(8)	118·7 (5)
C(7)—N(2)—C(8)	116·9 (4)	O(8)—C(14)—O(7)	119·3 (6)
O(8)—C(14)—C(13)	115·7 (6)	O(7)—C(14)—C(13)	125·0 (5)
C(3)—C(9)—C(10)	113·6 (4)	C(9)—C(10)—O(4)	126·0 (4)
C(9)—C(10)—O(5)	109·5 (4)	O(4)—C(10)—O(5)	124·4 (5)
C(10)—O(5)—C(11)	115·6 (4)	C(2)—C(12)—C(13)	116·2 (6)
C(14)—O(6)—C(15)	111·4 (6)	C(14)—C(13)—C(12)	115·4 (6)
O(1)—S(1)—N(1)—C(1)	−141·2 (3)	C(5)—S(1)—N(1)—C(4)	−87·4 (3)
O(1)—S(1)—N(1)—C(4)	28·9 (4)	N(1)—C(4)—C(6)—O(3)	63·5 (5)
O(2)—S(1)—N(1)—C(1)	−12·9 (4)	N(1)—C(4)—C(6)—N(2)	−121·0 (5)
O(2)—S(1)—N(1)—C(4)	157·3 (3)	C(3)—C(4)—C(6)—O(3)	−104·0 (5)
C(5)—S(1)—N(1)—C(1)	102·4 (3)	C(3)—C(4)—C(6)—N(2)	71·6 (6)

Experimental. The *N*-mesylpyrrole (I) was prepared *via* the mesylated acid from the parent benzylpyrrole carboxylate (De Voss, 1988). Transparent prismatic crystals were obtained by vapour diffusion from hexane/dichloromethane. Crystal 0·19 × 0·23 × 0·34 mm, Nicolet *R*3*mμ* diffractometer, graphite-monochromated Cu *Kα* radiation, cell parameters refined from diffractometer setting angles for 25 centred reflexions in the range $45 \le 2\theta \le 55°$. Intensity data collected by ω–2θ scans, scan range from 1·0° below α_1 to 1·0° above α_2 for 5605 reflexions ($5 \le 2\theta \le 116°$: h −16/16, k −16/0, l 0/21). Three standard reflexions (600, 040, 006) measured in every 100 showed no significant crystal decay. Of 2651 unique reflexions ($R_{\text{int}} = 0{\cdot}008$), 2258 with $F > 4\sigma(F)$ were used in the analysis. Structure solved by direct methods and refined by blocked cascade least squares based on F, with eight low angle reflections affected by extinction omitted. Anisotropic thermal parameters for non-H atoms; hydrogen atoms in calculated positions riding on C at 0·96 Å with separate overall U's for methyl-H and methylene-H. 229 parameters, $R = 0{\cdot}073$, $wR = 0{\cdot}090$ for 2250 observed data ($R = 0{\cdot}088$, $wR = 0{\cdot}1204$ for all 2651 unique data), $w^{-1} = [\sigma^2(F_o) + 0{\cdot}002F_o^2]$, $S = 2{\cdot}18$, $\Delta/\sigma = 0{\cdot}042$ (max.) and 0·009 (mean) $\Delta\rho$ within +0·59 and −0·36 e Å^{-3}. The peripheral atoms of the structure exhibit high thermal motion, which could not be interpreted in terms of disorder, but which reduces the precision of the analysis. The *SHELXTL* program system (Sheldrick, 1983) was used throughout with scattering factors taken from *International Tables for X-ray Crystallography* (1974).



(I)

Final atomic parameters are listed in Table 1* and molecular geometry is in Table 2. The molecular structure and atomic numbering are illustrated in Fig. 1.

Related literature. The geometry and conformation of the *N*-methylsulfonyl (*N*-mesyl) group are in good agreement with other examples reviewed by Kálmán, Párkányi & Schawartz (1977). Structural characteristics of the carboxylic amide group are reviewed by Chakrabarti & Dunitz (1982); the sterically-induced out-of-plane twist observed here is common in tertiary aryl carboxamides.

We thank the SERC for provision of equipment and for financial support to JJ De V. ICI Plant Protection Division and St John's College, Cambridge, are thanked for financial assistance to MJD.

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* Lists of structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51523 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

Acta Cryst. (1989). C**45**, 694–695

The structure of $[CdNi(CN)_4(C_6H_8N_2)(NH_3)]$: Corrigendum.

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(*Received* 19 *September* 1988; *accepted* 18 *October* 1988)

Abstract

The crystal structure of this compound, recently described in space group $Pna2_1$, has been more satisfactorily refined in $Pnma$. The final R is 0·019 for 104 parameters, compared to an R of 0·033 for 171 parameters in $Pna2_1$. The revised structure shows more reasonable interatomic distances involving the bridging C—N groups and a planar rather than a non-planar pyridine ring.

The structure of this polymeric compound, *catena*-poly-[tri-μ-cyano-(ammine)(2-amino-3-methylpyridine)cadmium-μ-cyano-nickel], has recently been described (Hökelek & Ülkü, 1988; hereinafter, HU) in the non-centrosymmetric space group $Pna2_1$ [orthorhombic; $a = 13·535$ (1), $b = 13·607$ (1), $c = 7·645$ (1) Å, $Z = 4$] and refined to an R of 0·033 for 1632 'observed' reflections. It is properly described in the centrosymmetric space group $Pnam$ (transformed here to the conventional $Pnma$, No. 62).

Initial coordinates in $Pnma$ were obtained from the values in Table 2 of HU by decrementing the z's by about 0·64 (so that most of the atoms lie at $z = 0·25$ or $z = 0·75$), interchanging y and z, and averaging N(1) with N(3), N(2) with N(4), C(1) with C(3), and C(2) with C(4). Least-squares refinement with anisotropic U_{ij}'s, including an extinction

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* Contribution No. 7852.