carboxyl groups are twisted in such a way that O(2) and O(3) are on the same side of the plane of the C atoms at distances of 0·122 (3) and 0·099 (3) Å respectively. This distortion from planarity is probably caused by a rather strong intramolecular hydrogen bond O(2)···O(3) of 2·403 (4) Å. A similar situation has been observed in other compounds containing hydrogen maleate residues, for example in manganese(II) hydrogen maleate tetrahydrate (Lis, 1983) and in imidazolium hydrogen maleate (Sakhawat Hussain, Schlemper & Fair, 1980; James & Matsushima, 1976).

The arrangement of C atoms around N in $N(CH_3)_4^+$ is almost ideally tetrahedral. The N—C lengths range from 1·483 (4) to 1·497 (4) Å, and the C—N—C angles from 108·6 (2) to 110·7 (2)° with average values of 1·489 (2) Å and 109·5 (1)° respectively.

There are no unusually close contacts between the $N(CH_3)_4^+$ ions and $C_4H_3O_4^-$ ions in the structure.

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2,6-Dimethyl-4*H*-1-thiapyran-4-one*

BY A. BANERJEE, C. J. BROWN AND P. C. JAIN†

Department of Metallurgy and Materials Engineering, City of London Polytechnic, Central House, Whitechapel High Street, London E1 7PF, England

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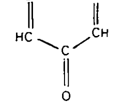
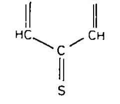
Abstract. C_7H_8OS, $M_r = 140·20$, monoclinic, $I2/a$, $a = 9·036$ (4), $b = 11·348$ (5), $c = 7·360$ (4) Å, $\beta = 104·4$ (1)°, $V = 730·99$ Å^3, $Z = 4$, $D_m = 1·275$ (10), $D_x = 1·274$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·35$ mm^{-1}, $F(000) = 296$, $T = 294$ K, $R = 0·028$ for 456 observed diffractometer reflexions. Molecules are effectively planar and stack in the unit cell in a close-packed arrangement in layers perpendicular to **c**, with the S, O and one C atom lying on twofold axes of symmetry. The S to C distance [1·737 (2) Å] represents a bond order of 1·5. There is no intermolecular bonding other than van der Waals with shortest distance 3·594 (4) Å.

Introduction. Research into the effectiveness of derivatives of γ-pyrones as corrosion inhibitors for steel led to the synthesis of the title compound (I) as a new intermediate. The crystal-structure determination was undertaken to afford a comparison with the corresponding homologous thiopyrone (II) (Toussaint, 1956).



(I)



(II)

Experimental. Material prepared by Drs E. M. Briggs and A. E. Hill of the Chemistry Department of this polytechnic (Hill, 1971). Sample recrystallized by vacuum sublimation to give white acicular crystals elongated along **c** (≈ 0·2 × 0·2 × 1·0 mm), m.p. 377 K. Density by flotation in NaI solution. Lattice parameters initially from Stoe Reciprocal Lattice Explorer and subsequently refined by least squares

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* 2,6-Dimethyl-4*H*-thiin-4-one.

† Present address: Department of Chemistry, Kurukshetra University, India.

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using 14 *hkl* reflexions on a Stoe Stadi-2 diffractometer. Crystals had a very limited life (<1 day) in the X-ray beam in air, so were coated with silicone grease and mounted in a sealed lithium borate glass 0·5 mm capillary, which prolonged their life to several weeks. Intensities on the diffractometer with crystal mounted parallel to **c**. $\theta_{\max} = 30°$, index range h −12 to +12, k 0 to 15, l 0 to 5; no measurable intensities on sixth layer. Six standard reflexions, no variation. 571 observed reflexions of which 115 had $I < 3\sigma(I)$. Data supplemented by a few visual Weissenberg photographic intensities for 00*l* and for $\theta < 5°$. Structure solved initially from a Patterson synthesis and subsequently confirmed by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) which failed for space group *I*2/*a*, but gave all nine non-H atoms in *Ia* using 136 terms ($E_{hkl} > 1{\cdot}2$), refined in *I*2/*a* [equivalent positions 0,0,0; $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ ± (x,y,z; $\frac{1}{2}+x$, $-y$, z)] by least squares on F using *NRC* programs (Ahmed, Hall, Pippy & Huber, 1970) on our VAX 11/750 computer. H atoms by calculation and included in structure-factor calculations with $B_{\rm iso} = 5{\cdot}0$ Å^2, but not refined, non-H atoms refined using $B_{\rm iso}$ initially and B_{ij} finally until all shifts $<0{\cdot}2\sigma$, residual $\Delta\rho$ in final difference Fourier map within $+0{\cdot}2$ e Å^{-3}, scattering factors from *International Tables for X-ray Crystallography* (1974), $R = 0{\cdot}028$, $wR = 0{\cdot}050$, $\sqrt{w} = 1/F_o$.

Discussion. Atomic coordinates and equivalent isotropic temperature factors are listed in Table 1,* bond lengths and angles in Table 2. The packing of the molecules in the unit cell is shown in Fig. 1. The overlap of the stacking of the molecules is closely similar to that found in the homologous thiopyrone (Toussaint, 1956) with the heterocyclic rings lying over each other rather than in a staggered arrangement.

The ring S atom is bonded to two C atoms at 1·737 (2) Å which corresponds to a bond order of 1·5 (Abrahams, 1956) assuming C—S (single) = 1·82 and (double) = 1·61 Å. This is in good agreement with 1·744 (3) Å in tetraphenyldithiapyranylidene (Luss & Smith, 1980) and the mean value of 1·734 (5) Å in 2,2′,6,6′-tetraethoxycarbonyl-4,4′-bithiapyranylidene (Darocha, Titus, Sandman & Warner, 1982). The angle C—S—C [109·1 (1)°] also agrees with 109° in di-*p*-tolyl sulphide (Blackmore & Abrahams, 1955).

The C—C and C—O bonds lie within the commonly found range of values in simple organic compounds, but are appreciably different from those found in γ-pyrone rings which are coordinated to metal atoms [see, *e.g.*, the averaged values in tris(2,6-dimethyl-4*H*-pyran-4-one)dinitratocadmium(II) (Banerjee, Brown, Jain & Gautam, 1984)]. The shortest intermolecular distance between non-H atoms is 3·594 (4) Å for C(3)—O (*a*-glide-related), and there are ten other contacts between non-H atoms less than 4·0 Å. The molecule is effectively planar with mean out-of-plane distance 0·006 Å.

Table 1. *Final atomic parameters with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}(\beta_{11}/a^{*2} + \beta_{22}/b^{*2} + \beta_{33}/c^{*2}).$$

	x	y	z	$B_{\rm eq}$(Å^2)
S	0·2500	0·8595 (1)	0·5000	1·51 (1)
O	0·2500	0·4741 (2)	0·5000	2·96 (4)
C(1)	0·5668 (3)	0·8257 (4)	0·5610 (4)	4·87 (6)
C(2)	0·4108 (2)	0·7707 (2)	0·5293 (4)	2·21 (4)
C(3)	0·3924 (3)	0·6553 (2)	0·5244 (4)	2·66 (4)
C(4)	0·2500	0·5788 (3)	0·5000	2·62 (5)

Table 2. *Bond lengths* (Å) *and interbond angles* (°)

S—C(2)	1·737 (2)	C(3)—C(4)	1·525 (3)
C(1)—C(2)	1·506 (4)	C(4)—O	1·189 (4)
C(2)—C(3)	1·320 (3)		
C(2)—S—C(2$^{\rm i}$)	109·1 (1)	C(2)—C(3)—C(4)	131·7 (2)
S—C(2)—C(1)	120·1 (2)	C(3)—C(4)—O	124·7 (2)
S—C(2)—C(3)	118·4 (2)	C(3)—C(4)—C(3$^{\rm i}$)	110·7 (2)
C(1)—C(2)—C(3)	121·5 (2)		

Symmetry code: (i) $\frac{1}{2} - x, y, 1 - z$.

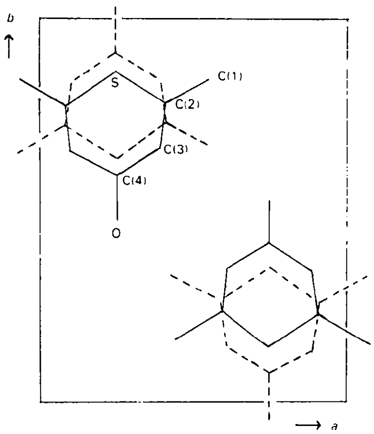

Fig. 1. Arrangement of molecules in projection of unit cell. The dashed lines represent molecules with $z \approx 0$, and the full lines those with $z \approx \frac{1}{2}$. The separation is $(c/2)\sin\beta = 3{\cdot}56$ Å.

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* Lists of structure factors, anisotropic temperature factors, calculated H-atom parameters, intermolecular distances and results of molecular-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42313 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The authors are grateful to a referee for pointing out a space-group discrepancy.

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Structure of a Benzamide Derivative

BY TOSHIO FURUYA* AND SUMIO IWANAMI

Central Research Laboratories, Yamanouchi Pharmaceutical Co. Ltd, Tokyo, Japan

AND AKIO TAKENAKA AND YOSHIO SASADA

Laboratory of Chemistry for Natural Products, Tokyo Institute of Technology, Nagatsuta 4259, *Midori-ku, Yokohama* 227, *Japan*

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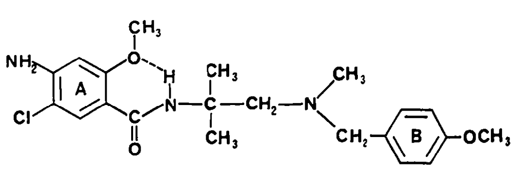
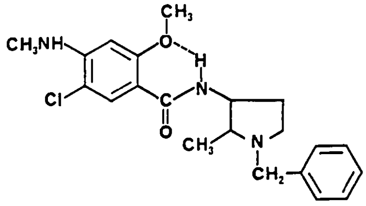
Abstract. 4-Amino-5-chloro-2-methoxy-*N*-{2-[*N*-(*p*-methoxybenzyl)methylamino]-1,1-dimethylethyl}benzamide, $C_{21}H_{28}ClN_3O_3$, $M_r = 405\cdot93$, monoclinic, $P2_1/c$, $a = 6\cdot144$ (1), $b = 26\cdot512$ (2), $c = 13\cdot741$ (2) Å, $\beta = 94\cdot76$ (1)°, $V = 2230\cdot5$ (5) Å^3, $Z = 4$, $D_m = 1\cdot21$, $D_x = 1\cdot209$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot71073$ Å, $\mu = 1\cdot92$ cm^{-1}, $F(000) = 864$, room temperature, $R = 0\cdot076$ for 3926 observed reflections. The benzamide moiety adopts a conformation and intramolecular hydrogen bond similar to those of a potent analogue, while the distance between the tertiary N atom and the center of the benzene ring is different. An intermolecular hydrogen bond of 3·019 (3) Å exists between the amino group N and the carbonyl O.

Introduction. The crystal-structure determination of the title compound (1) was undertaken as a part of continuous studies to find new potent neuroleptic drugs in benzamide derivatives in order to establish the three-dimensional structure–activity relationships. The crystal structure of a most potent compound, YM-09151-2 (2) (Furuya, Iwanami, Takenaka & Sasada, 1982), has been determined previously. Even though the present compound is not an active one contrary to expectation, the comparison of the structural features of active and inactive compounds would be important for further studies.



(1)



(2)

Experimental. Colorless plate crystals obtained from an acetone solution, density measured by flotation in a mixture of benzene and carbon tetrachloride. Crystal size 0·5 × 0·3 × 0·4 mm, Rigaku automated four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Accurate unit-cell constants determined using 40 high-angle reflections in range $20 < 2\theta < 30°$. Intensities measured for $2 < 2\theta < 50°$, $-7 \leq h \leq 7$,

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* To whom correspondence should be addressed.

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