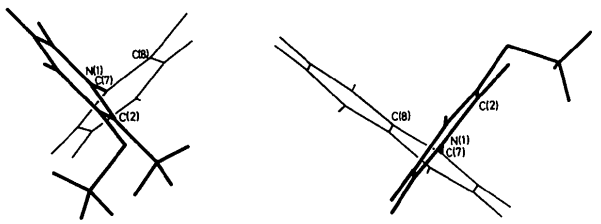

Compound (1) Compound (2)

Fig. 4. Twisted conformation along C—N bonds of *N*-aryl-4-pyridones with torsion angles −89·4 (4) (1) and −95 (1)° (2) for the atom sequence C(2)—N(1)—C(7)—C(8).

(1+*x*, *y*, *z*), O(4)···C(12), 3·359 (4) Å (−*x*+2, *y*+1, −*z*) (1) (Fig. 2), and O(4)···C(10), 3·261 (1) (*x*, *y*−1, *z*), O(4)···C(18), 3·32 (1) Å (*x*, *y*−1, *z*) (2) (Fig. 3). Molecules connected by C—H···O interactions are joined into two-dimensional sheets at $c \simeq 0$ and 1 (Fig. 2). In (2) molecules are aligned into chains running in the **b** direction (Fig. 3).

The crystals used in this structure determination were prepared by Dr M. Mintas, Faculty of Technology, University of Zagreb. Support of this research by the Self-Management Council for Scientific Research of S. R. Croatia is gratefully acknowledged.

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Structure of Lamotrigine Methanol Solvate: 3,5-Diamino-6-(2,3-dichlorophenyl)-1,2,4-triazine–Methanol, a Novel Anticonvulsant Drug

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Abstract. $C_9H_7Cl_2N_5.CH_3OH$, $M_r = 288$, monoclinic, $P2_1/n$, $a = 15{\cdot}456$ (3), $b = 11{\cdot}736$ (2), $c = 7{\cdot}300$ (3) Å, $\beta = 94{\cdot}417$ (3)°, $V = 1320{\cdot}3$ (5) Å^3, $Z = 4$, $D_x = 1{\cdot}449$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 42{\cdot}52$ cm^{-1}, $F(000) = 592$, $T = 293$ K, $R = 0{\cdot}055$ for 2444 observed reflections. The phenyl and triazine aromatic rings make a dihedral angle of 80·6 (9)° with each other. The bond linking the two rings is 1·480 (3) Å. The structure is stabilized by a network of hydrogen bonds involving amino and ring N atoms, one of the Cl atoms and the methanol of crystallization.

Introduction. Lamotrigine [3,5-diamino-6-(2,3-dichlorophenyl)-1,2,4-triazine] (I) is a novel anticonvulsant chemically unrelated to current antiepileptic drugs. Studies (Lamb, Leach, Miller & Wheatley, 1985) suggest its usefulness for generalized seizures with a possible wider profile than current drugs of first choice (*i.e.* phenytoin and carbamazepine). It may also be useful for absence seizures (Jawad, Oxley, Yuen & Richens, 1985). Clinical studies on humans (Cohen, Ashby, Crowley & Peck, 1985) showed that lamotrigine produced no important side effects and indicated a more favourable central-nervous-system (CNS) side-effect profile than phenytoin. In addition to lamotrigine's potential therapeutic utility as an antiepileptic, it is also possible that the drug, by virtue of its ability to block the release of excitatory amino acids, may prove of therapeutic value in several CNS degenerative disorders possibly attributable to the neurotoxic action of such acids, *e.g.* brain ischaemia and stroke, Huntington's chorea and senile dementia (Meldrum, 1985). The structure analysis reported here was undertaken as part of a study of convulsant and

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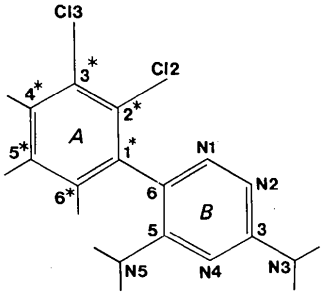
anticonvulsant compounds being carried out in this Department.



(I)

Experimental. Sample provided by the Wellcome Research Laboratories, UK. Colourless, prismatic crystal 0·3 × 0·2 × 0·6 mm mounted inside a glass capillary tube, with **c** parallel to the tube, used for data collection; preliminary Weissenberg and precession photographs yielded approximate cell dimensions and showed monoclinic (2/*m*) Laue symmetry. Space group $P2_1/n$ from systematic absences ($h0l$, $h+l=2n+1$; $0k0$, $k=2n+1$). Enraf–Nonius CAD-4 automated diffractometer, graphite monochromator, Cu $K\alpha$ radiation, 25 reflections ($25 \le \theta \le 28°$) used to obtain accurate cell dimensions by least-squares fit. Data collection by ω–2θ scan, scan width $(0{\cdot}90 + 0{\cdot}14\tan\theta)°$, vertical aperture = 4 mm, 2674 unique reflections measured ($-19 \le h \le 19$, $0 \le k \le 15$, $-9 \le l \le 9$), 2444 with $I \ge 3\sigma(I)$ ($1 \le \theta \le 75°$); three intensity standards ($33\bar{1}$, $0\bar{8}0$ and $\bar{3}\bar{3}5$) monitored at intervals of 200 reflections showed no significant variations during data collection; intensity data corrected for Lorentz–polarization factors; empirical absorption correction using a φ scan for the $00\bar{2}$ reflection (North, Phillips & Mathews, 1968) near $\chi = 90°$ measured at 10° intervals from $\varphi = 0$ to $\varphi = 360°$, normalized transmission factors 0·99 to 0·70. Structure solution by direct methods with *SHELX*76 (Sheldrick, 1976), atomic scattering factors from *SHELX*76; *E* map gave positions of the non-H atoms. Refinement by full-matrix least squares with anisotropic thermal factors for all the non-H atoms, isotropic for H atoms. H atoms located from difference synthesis except those of the methyl group which were placed in calculated positions riding on the pivotal C atom (C—H = 1·08 Å), function minimized was $w(|F_o| - |F_c|)^2$, $w = (\sigma^2|F_o| + 0{\cdot}0098|F_o|^2)^{-1}$, $R = 0{\cdot}055$, $wR = 0{\cdot}074$, max. (shift/σ) = 0·45. Final difference electron density synthesis showed residual electron density within −0·25 and +0·19 e Å^{-3}. Calculations carried out on Amdahl 470 V/8 computer. Geometrical calculations were performed with *XANADU* (Roberts & Sheldrick, 1975) and molecular illustrations were drawn with *PLUTO* (Motherwell & Clegg, 1978).

Table 1. *Atomic positional parameters and equivalent isotropic temperature factors for the non-*H *atoms with e.s.d.'s in parentheses*

$U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

	x	*y*	*z*	U_{eq}(Å^2)
C(1)*	0·7472 (1)	0·3743 (2)	0·7109 (3)	0·040 (11)
C(2)*	0·6864 (1)	0·4593 (2)	0·6662 (3)	0·039 (10)
C(3)*	0·6293 (1)	0·4931 (2)	0·7915 (3)	0·045 (12)
C(4)*	0·6326 (2)	0·4443 (3)	0·9649 (4)	0·057 (14)
C(5)*	0·6925 (2)	0·3600 (3)	1·0099 (4)	0·064 (17)
C(6)*	0·7499 (2)	0·3243 (2)	0·8826 (4)	0·055 (14)
Cl(2)	0·68300 (4)	0·52374 (5)	0·45293 (7)	0·050 (4)
Cl(3)	0·55150 (4)	0·59654 (7)	0·73782 (10)	0·062 (5)
N(1)	0·7920 (1)	0·2456 (2)	0·4806 (3)	0·046 (10)
N(2)	0·8445 (1)	0·2089 (2)	0·3539 (3)	0·049 (11)
C(3)	0·9151 (1)	0·2713 (2)	0·3333 (3)	0·042 (11)
N(4)	0·9387 (1)	0·3677 (1)	0·4231 (3)	0·041 (9)
C(5)	0·8855 (1)	0·4033 (2)	0·5468 (3)	0·038 (10)
C(6)	0·8085 (1)	0·3386 (2)	0·5759 (3)	0·038 (10)
N(3)	0·9679 (2)	0·2352 (2)	0·2050 (4)	0·058 (14)
N(5)	0·9038 (1)	0·4987 (2)	0·6396 (3)	0·054 (12)
C(1)	0·6057 (4)	0·0881 (6)	0·3809 (6)	0·112 (37)
O(1)	0·6332 (2)	0·1382 (2)	0·5504 (3)	0·068 (14)

* Atoms in the dichlorophenyl group.

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1.† Bond distances and angles are listed in Table 2. Fig. 1 shows a stereoview of the molecule, Fig. 2 illustrates the molecular packing.

The phenyl ring, *A*, of the dichlorophenyl moiety is planar. The equation of the least-squares plane defined by the six C atoms of ring *A* is $0{\cdot}656x' + 0{\cdot}693y' + 0{\cdot}298z' - 11{\cdot}881 = 0$, where x', y', z' are the coordinates in Å with respect to the orthogonal cell (Rollett, 1965). The r.m.s. displacement of the six atoms from the plane is 0·003 Å. The attached Cl atoms Cl(2) and Cl(3) and the C atom C(6) linking the triazine moiety are coplanar with ring *A* having deviations from the plane of 0·030, −0·041 and 0·014 Å respectively. These features are consistent with extensive electron delocalization within this group. No significant deviations from the average value of the bond lengths [1·386 (4) Å] or bond angles [120·0 (3)°] are observed for the phenyl ring. The triazine ring, *B*, is planar. The equation of the least-squares plane defined by the ring atoms is $0{\cdot}515x' - 0{\cdot}538y' + 0{\cdot}668z' - 6{\cdot}441 = 0$, with an r.m.s. atomic displacement of 0·003 Å. The attached amino N atoms N(3) and N(5) and the linking atom C(1)* are coplanar having deviations from the plane of −0·018, −0·023 and −0·005 Å respectively. All four C—N distances [average value 1·330 (3) Å] and the N—N distance in

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51338 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Molecular geometry*

E.s.d.'s are in parentheses.

(*a*) Bond lengths (Å)

C(1)*–C(2)*	1·391 (3)	N(4)–C(3)	1·343 (3)
C(2)*–C(3)*	1·378 (3)	N(2)–C(3)	1·333 (3)
C(3)*–C(4)*	1·387 (3)	N(1)–N(2)	1·347 (3)
C(4)*–C(5)*	1·378 (5)	N(1)–C(6)	1·308 (3)
C(5)*–C(6)*	1·398 (4)	N(3)–C(3)	1·356 (3)
C(1)*–C(6)*	1·382 (3)	N(5)–C(5)	1·328 (3)
C(1)*–C(6)	1·480 (3)	C(2)*–Cl(2)	1·728 (2)
C(5)–C(6)	1·441 (3)	C(3)*–Cl(3)	1·732 (2)
N(4)–C(5)	1·334 (3)	C(1)–O(1)	1·406 (5)

(*b*) Bond angles (°)

C(2)*–C(1)*–C(6)*	119·4 (2)	C(1)*–C(6)–C(5)	121·8 (2)
C(6)–C(1)*–C(6)*	120·5 (2)	C(1)*–C(6)–N(1)	118·8 (2)
C(6)–C(1)*–C(2)*	120·1 (2)	C(5)–C(6)–N(1)	119·4 (2)
C(1)*–C(2)*–C(3)*	120·4 (2)	C(6)–N(1)–N(2)	121·9 (2)
Cl(2)–C(2)*–C(1)*	119·9 (2)	N(1)–N(2)–C(3)	116·3 (2)
Cl(2)–C(2)*–C(3)*	119·7 (2)	N(2)–C(3)–N(4)	127·0 (2)
C(2)*–C(3)*–Cl(3)	121·4 (2)	N(4)–C(3)–N(3)	116·4 (2)
C(2)*–C(3)*–C(4)*	120·5 (2)	N(2)–C(3)–N(3)	116·6 (2)
C(4)*–C(3)*–Cl(3)	118·1 (2)	C(3)–N(4)–C(5)	115·8 (2)
C(3)*–C(4)*–C(5)*	119·4 (2)	C(6)–C(5)–N(4)	119·6 (2)
C(4)*–C(5)*–C(6)*	120·4 (3)	C(6)–C(5)–N(5)	121·0 (2)
C(5)*–C(6)*–C(1)*	119·9 (2)	N(4)–C(5)–N(5)	119·4 (2)

(*c*) Hydrogen bonds

D–H···*A*	*D*–H (Å)	H···*A* (Å)	*D*···*A* (Å)	*D*–H···*A* (°)
N(5)–H(51)···N(4^{iii})	0·987 (42)	1·983 (50)	2·960 (6)	170·1
N(5)–H(5)···O(1^{iii})	0·661 (37)	2·286 (61)	2·886 (7)	151·9
N(3)–H(3)···Cl(2^{iv})	0·902 (28)	2·776 (73)	3·538 (8)	143·0
O(1)–H(O1)···N(1^{ii})	0·781 (45)	2·121 (56)	2·839 (5)	153·0

(*d*) Intermolecular close contacts (Å)

N(1)	Cl(3^{iii})	3·518 (7)
N(2)	Cl(2^{iv})	3·126 (7)

Symmetry code: (i) x, y, z; (ii) $2-x, 1-y, 1-z$; (iii) $\frac{3}{2}-x, y+\frac{1}{2}, \frac{3}{2}-z$; (iv) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$.

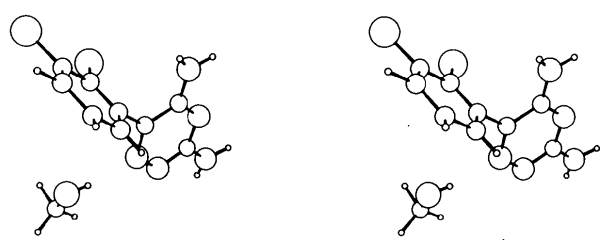


Fig. 1. Stereoview of the molecule along **c**.

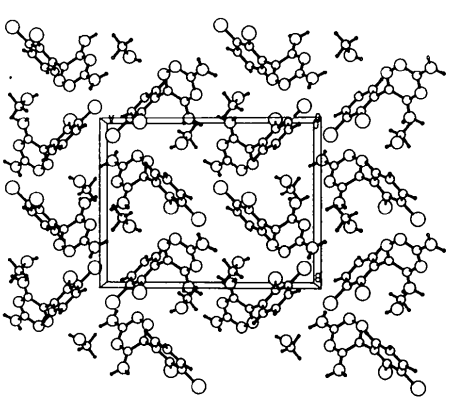


Fig. 2. The crystal structure viewed along **c**.

the triazine ring are intermediate between the expected single-bond lengths (1·47 and 1·45 Å respectively) and double-bond lengths (1·27 and 1·20 Å respectively). The N–N bond distance [1·348 (3) Å] in the triazine ring is longer than the mean value (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987) quoted for the N–N bond distance [1·304 (19) Å] in the pyridazine system, and the adjacent C(6)–N(1) distance [1·308 (3) Å] in the triazine ring is shorter than the corresponding mean [1·336 (14) Å] in pyridazine. The C(1)*–C(6) [1·480 (3) Å] linkage is short. The C–C distances are intermediate between normal single- and double-bond lengths. The two N–C linkages [average length 1·340 (3) Å] to the amino groups are also intermediate between the expected single- and double-bond lengths. All these features are consistent with extensive electron delocalization in this part of the molecule. The exocyclic angles at C(6) show slight asymmetry, C(1)*–C(6)–C(5) being 3° larger than C(1)*–C(6)–N(1). The dihedral angle between the phenyl plane and that defined for the triazine group is 80·6 (9)°. Orientation of the phenyl ring with respect to the triazine is governed by the torsion angles C(2)*–C(1)*–C(6)–N(1) and C(2)*–C(1)*–C(6)–C(5) which have values of −99·9 (3) and 80·1 (3)° respectively.

The molecules are linked by hydrogen bonds of four types. The nitrogen N(5) of the second amino group donates through H(51) to N(4) of the corresponding triazine ring forming a centrosymmetric hydrogen-bonded dimer, and through H(5) to the oxygen of the methanol of crystallization. The chlorine Cl(2) forms a hydrogen bond with the donor nitrogen N(3) of the first amino group. The methanol of crystallization forms an intramolecular hydrogen bond through H(O1) (donor) to N(1) of the triazine group. Details of the hydrogen bonding and close intermolecular contacts are given in Table 2.

A second form of lamotrigine has been crystallized from absolute ethanol having the following parameters in the monoclinic space group Aa: $a = 6·386$ (3), $b = 10·467$ (3), $c = 14·856$ (4) Å, $\beta = 100·774$ (3)°, $Z = 4$, $D_x = 1·736$ g cm^{-3}. A set of intensity data has been measured for this form, crystals of which are relatively unstable to X-rays, even when mounted in capillary tubes. Structure analysis is in progress.

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Novel Manganese(III) Oxidation Chemistry: X-ray Crystal Structure of 5,7,8-Trimethoxy-1-(2,4,5-trimethoxyphenyl)-1,2-dihydronaphthalene

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Abstract. $C_{22}H_{26}O_6$, $M_r = 386{\cdot}4$, monoclinic, $P2_1/c$, $a = 13{\cdot}021\ (4)$, $b = 11{\cdot}044\ (2)$, $c = 14{\cdot}036\ (9)$ Å, $\beta = 102{\cdot}53\ (4)°$, $V = 1970{\cdot}4$ Å^3, $Z = 4$, $D_x = 1{\cdot}30$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 0{\cdot}56$ cm^{-1}, $F(000) = 824$, $T = 293$ K, final $R = 0{\cdot}054$ for 1576 observed $[F_o \geq 5\sigma(F_o)]$ reflections. There is no crystallographically imposed symmetry. The title compound is prepared by manganese(III) acetate oxidative dimerization of two molecules of 2,4,5-trimethoxystyrene.

Introduction. During the course of our investigations on manganese(III) acetate mediated ring annulative approaches to the antineoplastic podophyllotoxins (Peterson, Do, Winter & Surjasasmita, 1988; Peterson, Do & Surjasasmita, 1988), an unusual oxidation product was obtained when the alkene component of the annulation reaction was 2,4,5-trimethoxystyrene. The single electron transfer oxidation (SETO) of electron-rich alkenes by manganese(III) recently had been reported by one of us to provide 1,2-diacetates, 1,2-hydroxyacetates, and their oxidation products (Fristad, Peterson, Ernst & Urbi, 1986). 2,4,5-Trimethoxystyrene was found to react by an entirely different pathway, however. Only products resulting from oxidative dimerization of this substrate, 5,7,8-trimethoxy-1-(2,4,5-trimethoxyphenyl)-1,2-dihydronaphthalene and some of the corresponding naphthalene, were obtained in the presence of manganese(III) acetate. This result was particularly surprising in view of the possibility for solvolysis of radical-cation intermediates in acetic acid. We wish to describe in this paper the X-ray crystal structure determination, synthesis, and spectral characterization of the title compound.

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Experimental. The title compound was prepared by reaction of a 343 K glacial acetic acid (35 mL) solution of 2,4,5-trimethoxystyrene (1·36 g, 7·0 mmol) and potassium acetate (0·69 g, 7·0 mmol) with manganese(III) acetate dihydrate (4·13 g, 2·2 mole equivalents of oxidant). The reaction was monitored for complete reduction of Mn^{III} to Mn^{II} with starch–potassium iodide test paper. Workup consisted of dilution with water, extraction with chloroform, and water washes of the combined extracts. Flash chromatography of the concentrate on silica gel using 40% ethyl acetate–hexane as eluent afforded 5,7,8-trimethoxy-1-(2,4,5-trimethoxyphenyl)-1,2-dihydronaphthalene† (544 mg) in 40% yield. Crystals (m.p. 430–431 K) suitable for X-ray analysis were obtained by recrystallization from chloroform–hexane. D_m not determined. Crystal 0·10 × 0·20 × 0·33 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$. Cell constants from setting angles of 25 reflections ($\theta > 18°$). Correction for Lorentz–polarization effect. $\theta_{max} = 50°$; h 0 to 15, k 0 to 13, l −16 to 16. Standard reflections observed every 3600 s of data collection time, 400; 060; 006. Variation = ±3%. 3748 reflections measured, 1576 independent observed reflections $[F_o \geq 5\sigma(F_o)]$. Structure solved utilizing *MULTAN*

† Physical data: IR (KBr) 2980, 2945, 2915, 2825, 1620 (CH=CH), 1590 (aromatic C=C), 1500 (aromatic C=C), 1490, 1460, 1450, 1390, 1335, 1320, 1305, 1225, 1195, 1165, 1100, 1030, 805, 785 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz) δ 6·82 (*dd*, $J = 9{\cdot}78$, 2·93 Hz, 1H, –CH=CH–CH_2–), 6·53 (*s*, 1H, aromatic H), 6·44 (*s*, 1H aromatic H), 6·30 (*s*, 1H aromatic H), 5·75–5·60 (*m*, 1H, –CH=CH–CH_2–), 4·88 (*d*, $J = 7{\cdot}68$ Hz, 1H, –CH–CH_2–), 3·91 (*s*, 3H, OCH_3), 3·87 (*s*, 6H, OCH_3), 3·85 (*s*, 3H, OCH_3), 3·57 (*s*, 3H, OCH_3), 3·39 (*s*, 3H, OCH_3), 2·77–2·55 (*m*, 1H, –CH–CH_2–), 2·42 (*dd*, $J = 17{\cdot}25$, 7·68 Hz, 1H, –CH–CH_2–). Analysis: calculated for $C_{22}H_{26}O_6$: C, 68·38; H, 6·78%; found: C, 68·35; H, 6·82%.