difference Fourier map and included in the refinement with isotropic thermal parameters. $\sum w(|F_o| - |F_c|)^2$ was minimized, $w = 4F_o^2/\sigma^2(F_o^2)$. Number of parameters refined = 214; final $R = 0.068$, $wR = 0.084$; $(\Delta/\sigma)_{max} = 0.05$, $S = 2.60$. The maximum and minimum peaks in the final difference Fourier map were 0.41 and -0.31 e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All numerical calculations were performed using the *TEXSAN* crystallographic software package of the Molecular Structure Corporation (1985). Final atomic parameters of non-H atoms are listed in Table 1; selected bond lengths, angles and hydrogen bonds are listed in Table 2.* A perspective view of 3-methoxytyramine perchlorate is shown in Fig. 1 with the atomic numbering scheme.

Related literature. 3-Methoxytyramine is the first metabolic product of dopamine; the crystal structure of the hydrochloride has been reported (Okabe, Mori & Sasaki, 1991). The amino side-chain orientation resembles those of catecholamines, all of which have the side chains oriented perpendicular to the phenyl ring plane [dopamine hydrochloride (Giesecke, 1980); adrenaline (Andersen, 1975); noradrenaline

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms, and coordinates and isotropic thermal parameters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55106 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0581]
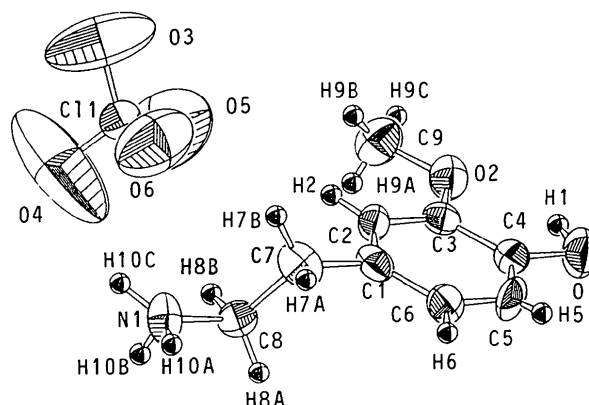

Fig. 1. Perspective view of 3-methoxytyramine perchlorate with the atomic numbering used.

hydrochloride (Carlström & Bergin, 1967)], but differs from that of 3-methoxytyramine hydrochloride in which the side chain lies in the same plane as the phenyl ring.

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Structure of an Acetone Solvate of 7,9-Diacetyl-2,5-dinitro-2,5,7,9-tetraazabicyclo[4.3.0]nonane

BY CLIFFORD GEORGE, RICHARD GILARDI AND JUDITH L. FLIPPEN-ANDERSON

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, DC 20375, USA

Abstract. $C_9H_{14}N_6O_6.\frac{1}{2}C_3H_6O$, $M_r = 331.29$, monoclinic, $C2/c$, $a = 29.512 (5)$, $b = 9.142 (1)$, $c = 10.872 (2)$ Å, $\beta = 92.46 (1)°$, $V = 2930.5 (8)$ Å^3, $Z = 8$, $D_x = 1.502$ Mg m^{-3}, $\lambda(Cu K\alpha) = 1.54184$ Å, $\mu = 1.04$ mm^{-1}, $F(000) = 1392$, $T = 295$ K, final $R = 0.043$, $wR = 0.047$ for 1708 independent observed reflections. Torsions and bends have destroyed the symmetry of this symmetrically substituted hetero-cycle. The torsion angles of the six-membered ring indicate a very distorted boat conformation, with the two N atoms at the bow and stern positions of the boat. The nitro-substituted N atoms are substantially pyramidalized, with bend angles between the N—N bonds and the adjacent CNC planes of 17.3 and 38.5°. The atoms of the five-membered ring fit a flattened envelope conformation, with an N atom

$$C_9H_{14}N_6O_6 \cdot \tfrac{1}{2}C_3H_6O$$

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{Å}^2 \times 10^3$)

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

	x	y	z	U_{eq}
C(1)	3715 (1)	7168 (3)	1579 (2)	31 (1)
N(2)	3331 (1)	6193 (2)	1364 (2)	36 (1)
C(3)	2981 (1)	6045 (3)	2257 (3)	41 (1)
C(4)	2909 (1)	7543 (3)	2830 (3)	42 (1)
N(5)	3048 (1)	8723 (2)	2020 (2)	37 (1)
C(6)	3535 (1)	8727 (3)	1815 (2)	33 (1)
N(7)	3786 (1)	9194 (2)	2908 (2)	36 (1)
C(8)	4021 (1)	8013 (3)	3564 (2)	38 (1)
N(9)	3996 (1)	6831 (2)	2671 (2)	33 (1)
N(10)	3363 (1)	5089 (3)	519 (2)	44 (1)
O(11)	3115 (1)	4031 (2)	635 (2)	60 (1)
O(12)	3622 (1)	5268 (2)	−317 (2)	55 (1)
N(13)	2765 (1)	8954 (3)	975 (2)	44 (1)
O(14)	2919 (1)	9691 (3)	143 (2)	66 (1)
O(15)	2380 (1)	8476 (2)	993 (2)	56 (1)
C(16)	3815 (1)	10586 (3)	3353 (2)	39 (1)
C(17)	3574 (1)	11769 (3)	2641 (3)	54 (1)
O(18)	4037 (1)	10812 (2)	4317 (2)	53 (1)
O(19)	4239 (1)	4729 (2)	1881 (2)	51 (1)
C(20)	4596 (1)	5490 (3)	3795 (3)	53 (1)
C(21)	4267 (1)	5630 (3)	2708 (2)	38 (1)
O(24)	5000	−975 (4)	2500	98 (2)
C(23)	5000	341 (6)	2500	60 (2)
C(22)	4802 (1)	1199 (4)	1443 (3)	83 (2)

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

C(1)—N(2)	1.454 (3)	C(1)—C(6)	1.545 (4)
C(1)—N(9)	1.452 (3)	N(2)—C(3)	1.454 (4)
N(2)—N(10)	1.371 (3)	C(3)—C(4)	1.522 (4)
C(4)—N(5)	1.463 (4)	N(5)—C(6)	1.466 (4)
N(5)—N(13)	1.396 (3)	C(6)—N(7)	1.438 (3)
N(7)—C(8)	1.454 (3)	N(7)—C(16)	1.362 (3)
C(8)—N(9)	1.452 (3)	N(9)—C(21)	1.359 (3)
N(10)—O(11)	1.224 (3)	N(10)—O(12)	1.223 (3)
N(13)—O(14)	1.230 (3)	N(13)—O(15)	1.218 (3)
C(16)—C(17)	1.493 (4)	C(16)—O(18)	1.228 (3)
O(19)—C(21)	1.220 (3)	C(20)—C(21)	1.502 (4)
O(24)—C(23)	1.203 (6)	C(23)—C(22)	1.490 (5)
C(23)—C(22)'	1.490 (5)		

N(2)—C(1)—C(6)	108.7 (2)	N(2)—C(1)—N(9)	114.5 (2)
C(6)—C(1)—N(9)	104.4 (2)	C(1)—N(2)—C(3)	121.4 (2)
C(1)—N(2)—N(10)	118.8 (2)	C(3)—N(2)—N(10)	116.9 (2)
N(2)—C(3)—C(4)	107.7 (2)	C(3)—C(4)—N(5)	111.7 (2)
C(4)—N(5)—C(6)	113.3 (2)	C(4)—N(5)—N(13)	115.4 (2)
C(6)—N(5)—N(13)	115.6 (2)	C(1)—C(6)—N(5)	111.7 (2)
C(1)—C(6)—N(7)	104.1 (2)	N(5)—C(6)—N(7)	110.4 (2)
C(6)—N(7)—C(8)	113.7 (2)	C(6)—N(7)—C(16)	126.4 (2)
C(8)—N(7)—C(16)	119.8 (2)	N(7)—C(8)—N(9)	102.4 (2)
C(1)—N(9)—C(8)	113.5 (2)	C(1)—N(9)—C(21)	120.7 (2)
C(8)—N(9)—C(21)	124.7 (2)	N(2)—N(10)—O(11)	116.9 (2)
N(2)—N(10)—O(12)	117.6 (2)	O(11)—N(10)—O(12)	125.3 (2)
N(5)—N(13)—O(14)	117.1 (2)	N(5)—N(13)—O(15)	117.2 (2)
O(14)—N(13)—O(15)	125.6 (2)	N(7)—C(16)—C(17)	118.1 (2)
N(7)—C(16)—O(18)	118.9 (2)	C(17)—C(16)—O(18)	123.0 (2)
N(9)—C(21)—O(19)	120.2 (2)	N(9)—C(21)—C(20)	116.9 (2)
O(19)—C(21)—C(20)	123.0 (3)	O(24)—C(23)—C(22)	121.8 (2)
O(24)—C(23)—C(22)'	121.8 (2)	C(22)—C(23)—C(22)'	116.5 (4)

lying 0.19 Å from the (± 0.01 Å) plane of the other four atoms. The two acetyl-substituted N atoms are essentially flat, with out-of-plane bends $< 10°$.

Experimental. A colorless laminar $0.07 \times 0.30 \times 0.32$ mm data crystal was provided by Clifford L. Coon of the Livermore National Laboratory, Livermore, California. An automated Siemens $R3m/V$ diffractometer with incident-beam monochromator was used for data collection. 25 centered reflections within $30 \leq 2\theta \leq 59°$ were used for determining lattice parameters. $(\sin\theta/\lambda)_{max} = 0.547 \text{ Å}^{-1}$, range of hkl: $-32 \leq h \leq 8$, $-10 \leq k \leq 0$, $-11 \leq l \leq 11$. Three standard reflections ($0\bar{2}3$, $0\bar{4}0$, $10,0,0$) monitored every 97 reflections showed random variation of 2.5% over data collection: $\theta/2\theta$-scan mode, scan width $[2\theta(K\alpha_1) - 0.90]$ to $[2\theta(K\alpha_2) + 0.90]°$, ω-scan rate a function of count rate (minimum 3.0, maximum $15.0° \text{ min}^{-1}$). 2953 reflections were measured, of which 2005 were unique ($R_{int} = 2.3\%$) and 1708 were considered observed with $F_o > 3\sigma(F_o)$. Data were corrected for Lorentz and polarization effects, but not for absorption. The structure solution, by direct methods, and the full-matrix least-squares refinement (on F) used programs in *SHELXTL80* (Sheldrick, 1980). The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0.000225$. Secondary-extinction parameter $p = 0.0009$ (1) in $F_c^* = F_c/[1.0 + 0.002(p)F_o^2/\sin(2\theta)]^{0.25}$. 234 parameters were refined: atomic coordinates for all atoms except methyl H atoms, anisotropic thermal parameters for non-H atoms. Methyl H atoms were included using a riding model (coordinate shifts of C applied to attached H atoms, C—H distance set to 0.96 Å, H angles idealized); fixed U_{iso} for all H atoms set to $1.1 U_{eq}(C)$ or, if methyl, $1.2 U_{eq}(C)$. $(\Delta/\sigma)_{max} = 0.01$, ratio of observations to parameters $= 7.3:1$, $R = 0.043$ ($R = 0.052$ for all data), $wR = 0.047$, $S = 1.76$. Final difference Fourier excursions were 0.29 and -0.23 e Å^{-3}. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV).† Atom numbering for Tables 1, atom coordinates, and 2, bond distances and angles, follows that shown in Fig. 1.
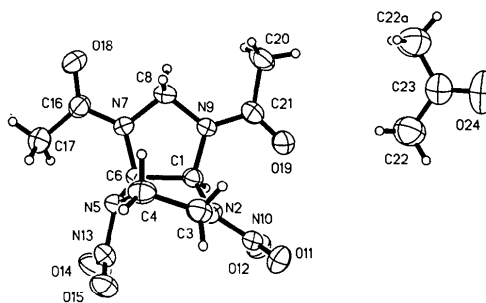

Fig. 1. A thermal ellipsoid plot of 7,9-diacetyl-2,5-dinitro-2,5,7,9-tetraazabicyclo[4.3.0]nonane and the closest neighboring acetone molecule, with ellipsoids drawn at the 20% probability level. The carbonyl bond of the acetone coincides with a twofold axis of the crystal.

Related literature. Two similar tetraazabicyclo-nonanes were reported by Flippen-Anderson, George & Gilardi (1990).

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2-Chloro-2'-methoxyacetophenone and 2-Chloro-4'-methoxyacetophenone

BY KEVIN L. EVANS, FRANK R. FRONCZEK AND RICHARD D. GANDOUR*

*Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, USA*

Abstract. 2-Chloro-2'-methoxyacetophenone (I), $C_9H_9ClO_2$, $M_r = 184.6$, monoclinic, $P2_1/c$, $a = 8.1261$ (11), $b = 14.4215$ (9), $c = 7.6839$ (6) Å, $\beta = 103.173$ (9)°, $V = 876.8$ (3) Å^3, $Z = 4$, $D_x = 1.399$ g cm^{-3} at 296 K, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 35.5$ cm^{-1}, $F(000) = 384$, 1750 unique data measured, final $R = 0.035$ for 1297 reflections with $I > 3.0\sigma(I)$. 2-Chloro-4'-methoxyacetophenone (II), $C_9H_9ClO_2$, $M_r = 184.6$, monoclinic, $P2_1/c$, $a = 7.6079$ (7), $b = 12.296$ (2), $c = 9.9240$ (13) Å, $\beta = 111.097$ (9)°, $V = 866.1$ (2) Å^3, $Z = 4$, $D_x = 1.416$ g cm^{-3} at 295 K, $\lambda(\text{Cu } K\alpha) = 1.54184$ Å, $\mu = 35.9$ cm^{-1}, $F(000) = 384$, 1782 unique data measured, final $R = 0.040$ for 1411 reflections with $I > 3.0\sigma(I)$. Maximum deviation from planarity of the aromatic ring is 0.005 (2) Å in (I) and 0.009 (2) Å in (II). In both (I) and (II) the methoxy substituent is nearly coplanar with the benzenoid ring with C—C—O—C torsion angles of −4.2 (2)° in (I) and −5.5 (3)° in (II). The C—Cl distance [1.779 (2) Å] in (I) is indistinguishable from that [1.772 (2) Å] in (II). The close CH$_3$O···CCl intramolecular contact [2.676 (2) Å] and the nearly linear angle [160.7 (1)°] for CH$_3$O···C—Cl in (I) suggest predisposition for an intramolecular nucleophilic substitution. The lack of dramatic change in the C—Cl distance in (I) discounts any strong interaction between the O atom and C—Cl bond.

Experimental. Data for both structures were obtained on an Enraf–Nonius CAD-4 diffractometer with a graphite monochromator using Cu $K\alpha$ radiation, from colorless crystals mounted in capillaries. The ω–2θ scans were designed for $I = 50\sigma(I)$, subject to maximum scan time of 120 s. Data reduction included corrections for background, Lorentz and polarization effects. Standard reflections exhibited only random fluctuations in intensity: ± 1.3% for (I) and ± 1.2% for (II). Absorption corrections were based on ψ scans. The structure of (I) was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Compound (II) is isomorphous with the bromo analog (Fronczek, Fouad & Gandour, 1991) and coordinates from that structure were used as a beginning refinement model. Refinements were carried out by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using Enraf-Nonius *SDP/VAX* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974) and anomalous coefficients of Cromer (1974). Non-H atoms were refined anisotropically; H atoms were located in difference maps and refined isotropically.

(I)   (II)
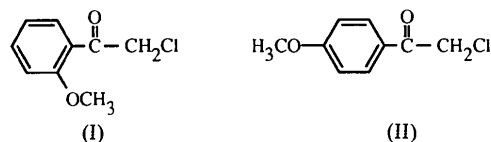
A colorless crystal of compound (I) was grown by evaporation of benzene from the crude reaction product of 2'-methoxyacetophenone with phosphorus pentachloride and phosphorus trichloride. Details of the data collection and structural refinement are given in Table 1. Coordinates† and

* To whom correspondence should be addressed.