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The dipolar cycloaddition of methyl acrylate to 1,5,6-trimethyl-3-oxidopyrazinium

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5,6-Dimethylpyrazin-2-one reacts with iodomethane to give a quaternary salt, deprotonation of which liberates a 3-oxidopyrazinium which undergoes a 1,3-dipolar cycloaddition with methyl acrylate to form methyl 5,8-dimethyl-4-methylene-2-oxo-3,8-diazabicyclo[3.2.1]octane-6-*exo*-carboxylate, $C_{11}H_{16}N_2O_3$, as the major product.

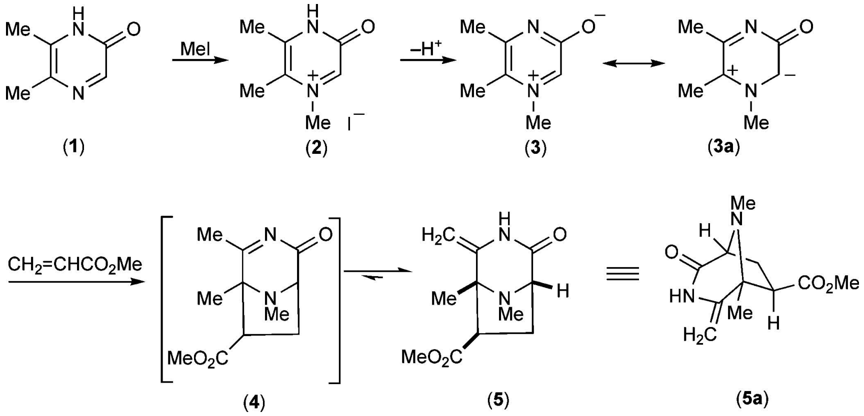
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.031
wR factor = 0.089
Data-to-parameter ratio = 8.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

We have been investigating the 1,3-dipolar cycloaddition chemistry of 3-oxidopyraziniums (Kiss *et al.*, 1987; Allway *et al.*, 1990; Yates *et al.*, 1995). These reactions efficiently produce bridged bicyclic systems, *viz*. 3,8-diazabicyclo[3.2.1]octanes, which comprise key structural components of such biologically active natural products as anticancer quinocarcin (Takahashi & Tomita 1983; Tomita *et al.*, 1983; Hirayama & Shirahata, 1983) and antibiotic lemonomycin (He *et al.*, 2000). Our studies were initially inspired by the series of benchmark papers by Katritzky and co-workers [for reviews, see Dennis *et al.* (1976) and Katritzky & Dennis (1989)] on the cycloadditions of 3-oxidopyridiniums. In neither Katritzky's extensive studies nor our own on 3-oxidopyraziniums had the possible influence of a substituent on the 1,3-dipole at one (or both) of the future ring-junction positions been assessed. This report describes our first study to remedy this omission, in which the reactivity of 1,5,6-trimethyl-3-oxidopyrazinium, (3) (see scheme; synthesis of 1,5,6-trimethyl-3-oxidopyrazinium and its reaction with methyl acrylate), was assessed.



5,6-Dimethylpyrazin-2-one, (1) (Jones, 1949; Karmas & Spoerri, 1952), was reacted with iodomethane to produce the methiodide (2), treatment of which with triethylamine allowed the generation of the zwitterion (3), *in situ* and in the presence of methyl acrylate. The reactivity and regioselectivity of such 3-oxidodiaziniums is easily understood in terms of a resonance contributor [(3a) in this case]. The immediate products of the

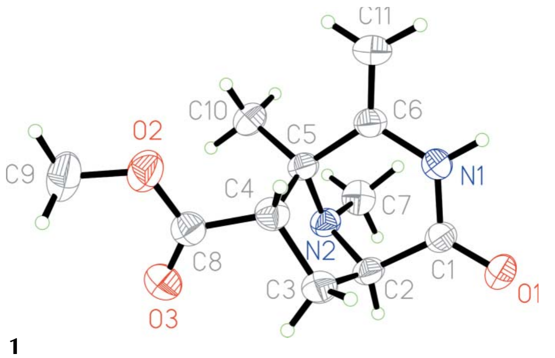


Figure 1
Plot of (4), with displacement ellipsoids drawn at the 50% probability level.

cycloadditions [(4) in this case] are not isolated, but tautomerize to the enamide structure [(5) in this case], (5a) showing better the bicyclic nature of the product. A mixture of two isomeric products was formed from which the major isomer was isolated, crystalline, allowing an X-ray analysis to show that it was the *exo*-ester (4) (Fig. 1). Thus, the additional oxidopyrazinium-6-methyl, appearing at the ring junction (C-5) in the cycloadduct, did not affect the efficiency or stereoselectivity of the cycloaddition, compared with the comparable reaction of 1,5-dimethyl-3-oxidopyrazinium which also gave a 6-*exo*-ester as the major product (Yates *et al.*, 1995).

Experimental

5,6-Dimethylpyrazin-2-one (Jones, 1949; Karmas & Spoerri, 1952) (800 mg, 6.5 mmol) and iodomethane (2 ml, 32.5 mmol, 5 equivalents) were heated under reflux in MeCN (150 ml) under nitrogen for 24 h. The solvent was evaporated under vacuum and the residue extracted with CH_2Cl_2. Insoluble material was removed by filtration and the solution evaporated, leaving 3,4-dihydro-1,5,6-trimethyl-3-oxopyrazinium iodide as a dark-brown crystalline solid (1.12 g, 66%; m.p. >523 K); ^{1}H NMR (D_2O, 300 MHz, δ, p.p.m.)): 8.20 (1H, *s*, C2—H), 4.15 (3H, *s*, NMe), 2.50 and 2.45 (2 × *s*, 2 × 3H, 2 × CMe). Analysis found: C 32.31, H 4.02, N 10.39%; $C_7H_{11}N_2O$ requires: C 31.60, H 4.17, N 10.53%

A solution of 3,4-dihydro-1,5,6-trimethyl-3-oxopyrazinium iodide (1.5 g, 5.6 mmol), Et_3N (1.6 ml, 11.2 mmol, 2 equivalents), and methyl acrylate (1.52 ml, 16.8 mmol, 3 equivalents) in dry MeCN (100 ml) was heated under reflux for 2 h. Solvents were removed from the resulting orange solution under vacuum, H_2O (30 ml) was added and the product extracted into CH_2Cl_2 (3 × 30 ml). The combined dried extract was evaporated leaving a brown oil (0.88 g, 70%) from which, by careful chromatography over silica, eluting with *n*-hexane–EtOAc (1:1), the major (thin-layer chromatography) adduct was obtained as colourless plates (330 mg, 26%; m.p. 383–388 K).

Crystal data

$C_{11}H_{16}N_2O_3$
$M_r = 224.26$
Monoclinic, $P2_1/c$
$a = 9.818$ (10) Å
$b = 7.89$ (2) Å
$c = 14.80$ (3) Å
$\beta = 102.23$ (8)°
$V = 1120$ (4) Å^3
$Z = 4$

$D_x = 1.330$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 17414 reflections
$\theta = 2.1$–25.0°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Plate, colourless
0.3 × 0.2 × 0.1 mm

Data collection

Rigaku R-AXIS II diffractometer
φ scans
Absorption correction: none
17414 measured reflections
1767 independent reflections
1516 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.037$
$\theta_{\rm max} = 25.0°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 9$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.089$
$S = 1.03$
1767 reflections
210 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.1608P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.037 (6)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1$^{\rm i}$	0.913 (18)	2.082 (19)	2.980 (6)	167.6 (15)

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

H atoms were found by difference Fourier methods and refined isotropically, with refined C—H distances in the range 0.932 (15)–1.041 (19) Å and an N—H distance of 0.913 (18) Å.

Data collection: *MSC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *DENZO* (Otwinowski & Minor, 1987); data reduction: *DENZO*; program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *TEXSAN* (Molecular Structure Corporation, 1995).

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methyl 8-methyl-4-methylene-2-oxo-3,8-diazabicyclo[3.2.1]octane-6-*exo*-carboxylate

Crystal data

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$F(000) = 480$
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Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 17414 reflections
$\theta = 2.1$–$25.0°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Plate, colourless
0.3 × 0.2 × 0.1 mm

Data collection

Rigaku R-AXIS diffractometer
Radiation source: Rigaku rotating anode
Graphite monochromator
116 × 3° φ scans
17414 measured reflections
1767 independent reflections
1516 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$
$\theta_{\rm max} = 25.0°$, $\theta_{\rm min} = 2.1°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 9$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.089$
$S = 1.03$
1767 reflections
210 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.1608P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.13$ e Å^{-3}
Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.037 (6)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.49890 (11)	0.24550 (13)	0.76055 (8)	0.0498 (3)
O2	1.10556 (10)	0.28198 (14)	0.63198 (8)	0.0523 (3)
O3	0.99596 (11)	0.53179 (13)	0.62400 (8)	0.0519 (3)
N1	0.62845 (13)	0.06832 (15)	0.69294 (8)	0.0394 (3)
H1	0.5847 (18)	−0.021 (2)	0.7137 (12)	0.058 (5)*
N2	0.67389 (11)	0.32417 (14)	0.57739 (7)	0.0347 (3)
C1	0.58496 (14)	0.22418 (17)	0.71177 (9)	0.0360 (4)
C2	0.65104 (15)	0.36832 (17)	0.66899 (9)	0.0367 (4)
H2	0.5926 (16)	0.469 (2)	0.6655 (10)	0.043 (4)*
C3	0.79972 (16)	0.3988 (2)	0.72487 (11)	0.0446 (4)
H3A	0.8088 (16)	0.369 (2)	0.7904 (13)	0.053 (4)*
H3B	0.8251 (18)	0.517 (2)	0.7213 (12)	0.056 (5)*
C4	0.89119 (14)	0.28417 (18)	0.67810 (10)	0.0370 (4)
H4	0.9360 (14)	0.2009 (19)	0.7184 (10)	0.036 (4)*
C5	0.78439 (13)	0.19369 (16)	0.59858 (9)	0.0336 (3)
C6	0.72950 (14)	0.03561 (16)	0.63962 (9)	0.0348 (3)
C7	0.54837 (17)	0.2728 (2)	0.50992 (11)	0.0467 (4)
H7A	0.4741 (18)	0.362 (2)	0.5114 (11)	0.058 (5)*
H7B	0.5689 (18)	0.261 (2)	0.4480 (14)	0.059 (5)*
H7C	0.5091 (17)	0.160 (2)	0.5271 (12)	0.062 (5)*
C8	1.00029 (14)	0.38310 (19)	0.64213 (9)	0.0386 (4)
C9	1.21255 (18)	0.3595 (3)	0.59168 (15)	0.0615 (5)
H9A	1.1729 (19)	0.391 (2)	0.5257 (15)	0.074 (6)*
H9B	1.250 (2)	0.452 (3)	0.6286 (16)	0.079 (6)*
H9C	1.289 (3)	0.273 (3)	0.5965 (16)	0.094 (7)*
C10	0.83963 (18)	0.1504 (2)	0.51235 (11)	0.0470 (4)
H10A	0.8685 (17)	0.257 (2)	0.4818 (12)	0.054 (4)*
H10B	0.9270 (19)	0.078 (2)	0.5311 (12)	0.065 (5)*
H10C	0.7628 (18)	0.087 (2)	0.4651 (13)	0.062 (5)*
C11	0.76950 (18)	−0.12241 (19)	0.63052 (12)	0.0500 (4)
H11A	0.8372 (17)	−0.147 (2)	0.5951 (12)	0.053 (4)*
H11B	0.7307 (19)	−0.215 (2)	0.6613 (13)	0.062 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0585 (7)	0.0439 (6)	0.0569 (7)	0.0033 (5)	0.0350 (6)	0.0006 (5)
O2	0.0375 (6)	0.0560 (7)	0.0653 (7)	0.0066 (5)	0.0154 (5)	0.0031 (5)
O3	0.0581 (7)	0.0384 (7)	0.0619 (7)	−0.0046 (5)	0.0189 (5)	0.0011 (5)
N1	0.0489 (7)	0.0294 (7)	0.0450 (7)	0.0020 (5)	0.0215 (6)	0.0055 (5)
N2	0.0397 (6)	0.0314 (6)	0.0348 (6)	0.0048 (5)	0.0121 (5)	0.0027 (5)
C1	0.0410 (8)	0.0336 (8)	0.0358 (7)	0.0041 (6)	0.0138 (6)	0.0006 (6)
C2	0.0453 (8)	0.0285 (8)	0.0404 (8)	0.0070 (6)	0.0182 (6)	0.0006 (6)
C3	0.0544 (9)	0.0413 (9)	0.0417 (9)	−0.0064 (7)	0.0184 (7)	−0.0105 (7)
C4	0.0402 (8)	0.0342 (8)	0.0361 (8)	0.0023 (6)	0.0070 (6)	0.0032 (6)
C5	0.0379 (8)	0.0292 (7)	0.0359 (7)	0.0036 (5)	0.0127 (6)	−0.0010 (5)
C6	0.0379 (7)	0.0308 (8)	0.0362 (7)	0.0032 (6)	0.0090 (6)	−0.0012 (5)
C7	0.0453 (9)	0.0505 (10)	0.0425 (9)	0.0030 (7)	0.0052 (7)	0.0063 (7)
C8	0.0373 (8)	0.0407 (9)	0.0365 (7)	−0.0001 (6)	0.0049 (6)	−0.0027 (6)
C9	0.0384 (9)	0.0866 (15)	0.0620 (12)	−0.0051 (10)	0.0164 (8)	−0.0053 (11)
C10	0.0529 (10)	0.0485 (10)	0.0452 (9)	0.0005 (8)	0.0231 (8)	−0.0074 (7)
C11	0.0575 (10)	0.0318 (9)	0.0642 (10)	0.0053 (7)	0.0212 (8)	−0.0024 (7)

Geometric parameters (Å, º)

O1—C1	1.234 (2)	C4—C5	1.572 (3)
O2—C8	1.339 (2)	C4—H4	0.932 (15)
O2—C9	1.448 (3)	C5—C10	1.527 (3)
O3—C8	1.202 (3)	C5—C6	1.534 (3)
N1—C1	1.349 (3)	C6—C11	1.322 (4)
N1—C6	1.416 (2)	C7—H7A	1.018 (18)
N1—H1	0.913 (18)	C7—H7B	0.983 (19)
N2—C2	1.462 (3)	C7—H7C	1.026 (19)
N2—C7	1.469 (3)	C9—H9A	1.00 (2)
N2—C5	1.480 (3)	C9—H9B	0.94 (2)
C1—C2	1.513 (3)	C9—H9C	1.01 (2)
C2—C3	1.537 (3)	C10—H10A	1.025 (18)
C2—H2	0.972 (16)	C10—H10B	1.018 (19)
C3—C4	1.538 (3)	C10—H10C	1.041 (19)
C3—H3A	0.982 (18)	C11—H11A	0.950 (17)
C3—H3B	0.973 (18)	C11—H11B	0.978 (19)
C4—C8	1.510 (3)		

C8—O2—C9	116.07 (19)	N2—C5—C4	100.71 (16)
C1—N1—C6	124.80 (12)	C10—C5—C4	115.36 (15)
C1—N1—H1	116.3 (11)	C6—C5—C4	107.89 (16)
C6—N1—H1	118.8 (11)	C11—C6—N1	119.13 (14)
C2—N2—C7	115.11 (15)	C11—C6—C5	126.09 (18)
C2—N2—C5	102.63 (13)	N1—C6—C5	114.77 (15)
C7—N2—C5	115.08 (16)	N2—C7—H7A	107.2 (9)
O1—C1—N1	122.07 (13)	N2—C7—H7B	110.5 (10)

O1—C1—C2	123.38 (16)	H7A—C7—H7B	111.9 (14)
N1—C1—C2	114.55 (18)	N2—C7—H7C	112.1 (10)
N2—C2—C1	111.89 (16)	H7A—C7—H7C	106.9 (14)
N2—C2—C3	102.78 (14)	H7B—C7—H7C	108.2 (14)
C1—C2—C3	109.57 (14)	O3—C8—O2	123.22 (16)
N2—C2—H2	110.5 (9)	O3—C8—C4	126.06 (14)
C1—C2—H2	109.4 (9)	O2—C8—C4	110.72 (19)
C3—C2—H2	112.6 (9)	O2—C9—H9A	109.9 (11)
C2—C3—C4	104.17 (15)	O2—C9—H9B	108.4 (13)
C2—C3—H3A	111.8 (9)	H9A—C9—H9B	113.3 (18)
C4—C3—H3A	111.4 (10)	O2—C9—H9C	106.3 (13)
C2—C3—H3B	110.2 (10)	H9A—C9—H9C	111.5 (18)
C4—C3—H3B	111.1 (10)	H9B—C9—H9C	107.1 (18)
H3A—C3—H3B	108.2 (14)	C5—C10—H10A	111.4 (10)
C8—C4—C3	112.36 (19)	C5—C10—H10B	109.0 (10)
C8—C4—C5	112.40 (16)	H10A—C10—H10B	106.5 (14)
C3—C4—C5	104.07 (15)	C5—C10—H10C	109.5 (10)
C8—C4—H4	108.4 (8)	H10A—C10—H10C	109.1 (14)
C3—C4—H4	111.5 (9)	H10B—C10—H10C	111.3 (14)
C5—C4—H4	108.0 (9)	C6—C11—H11A	120.2 (10)
N2—C5—C10	110.64 (14)	C6—C11—H11B	120.1 (11)
N2—C5—C6	110.23 (16)	H11A—C11—H11B	119.7 (15)
C10—C5—C6	111.47 (16)		

C6—N1—C1—O1	−178.22 (13)	C8—C4—C5—N2	−92.40 (16)
C6—N1—C1—C2	1.3 (2)	C3—C4—C5—N2	29.43 (13)
C7—N2—C2—C1	57.34 (16)	C8—C4—C5—C10	26.72 (18)
C5—N2—C2—C1	−68.45 (18)	C3—C4—C5—C10	148.56 (14)
C7—N2—C2—C3	174.81 (12)	C8—C4—C5—C6	152.07 (12)
C5—N2—C2—C3	49.02 (16)	C3—C4—C5—C6	−86.09 (18)
O1—C1—C2—N2	−144.21 (14)	C1—N1—C6—C11	176.04 (14)
N1—C1—C2—N2	36.25 (18)	C1—N1—C6—C5	−2.9 (2)
O1—C1—C2—C3	102.47 (18)	N2—C5—C6—C11	149.07 (16)
N1—C1—C2—C3	−77.07 (18)	C10—C5—C6—C11	25.8 (2)
N2—C2—C3—C4	−28.60 (15)	C4—C5—C6—C11	−101.83 (19)
C1—C2—C3—C4	90.5 (2)	N2—C5—C6—N1	−32.04 (17)
C2—C3—C4—C8	120.99 (15)	C10—C5—C6—N1	−155.31 (13)
C2—C3—C4—C5	−0.87 (15)	C4—C5—C6—N1	77.05 (17)
C2—N2—C5—C10	−171.11 (11)	C9—O2—C8—O3	−3.8 (2)
C7—N2—C5—C10	63.08 (18)	C9—O2—C8—C4	175.82 (13)
C2—N2—C5—C6	65.14 (15)	C3—C4—C8—O3	−22.0 (2)
C7—N2—C5—C6	−60.67 (19)	C5—C4—C8—O3	94.99 (18)
C2—N2—C5—C4	−48.63 (14)	C3—C4—C8—O2	158.37 (13)
C7—N2—C5—C4	−174.43 (11)	C5—C4—C8—O2	−84.64 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.913 (18)	2.082 (19)	2.980 (6)	167.6 (15)

Symmetry code: (i) −*x*+1, *y*−1/2, −*z*+3/2.