

type, piled up along the [110] direction. It can be described as mixed stacks of alternating organic and inorganic dimers (Fig. 2).

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Structure of Dicarboxylbis(η^2 -dimethyl 7-acetyl-7-azabicyclo[2.2.1]hept-2-ene-2,3-dicarboxylate- κN)molybdenum(0)

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(Received 13 November 1990; accepted 18 June 1991)

Abstract. $[\text{Mo}(\text{C}_{24}\text{H}_{34}\text{N}_2\text{O}_{10})(\text{CO})_2]$, $M_r = 662.51$, monoclinic, $C2/c$, $a = 61.3502$ (41), $b = 10.4370$ (4), $c = 17.7387$ (14) Å, $\beta = 99.742$ (6)°, $V = 11194.5$ (18) Å³, $Z = 16$, $D_x = 1.57$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54056$ Å, $\mu = 44.7$ cm⁻¹, $F(000) = 5472$, $T = 298$ K, $R = 0.046$ for 7459 reflections with $I > 3\sigma(I)$. There are two independent molecules per unit cell. In each molecule, two CO groups are *cis* to each other with Mo—CO distances from 1.955 (5) to 1.982 (7) Å. Two substituted double bonds are bound to the Mo atom [Mo—C from 2.193 (6) to 2.238 (6) Å]. The C—C distances for bound and non-bound double bonds are 1.437 (8) and 1.34 (1) Å, respectively. The Mo—N distances are 2.336 (4), 2.347 (5), 2.361 (5) and 2.339 (5) Å.

Experimental. The title compound, $\text{C}_{26}\text{H}_{34}\text{MoN}_2\text{O}_{12}$, was synthesized as reported by Sun & Chow (1988). Yellow crystals were grown from Et_2O . A suitable prism crystal with dimensions $0.15 \times 0.20 \times 0.30$ mm was mounted on a glass fiber with epoxy resin. Cell constants were derived from least-squares refinement of 25 reflections having $50 < 2\theta < 52^\circ$. Intensity data were collected at room temperature using the $\theta/2\theta$ scanning technique on an Enraf-Nonius CAD-4 diffractometer with monochromated $\text{Cu } K\alpha$ radiation. A total of 9595 reflections were measured with $0 <$

$2\theta < 130^\circ$ ($h = -72$ to 70 , $k = 0$ to 12 , $l = 0$ to 20), which were averaged to 9305 unique reflections with $R_{\text{int}} = 0.019$. Three standard reflections ($17\bar{3}6$, $1\bar{7}3\bar{6}$, $\bar{6}47$) were measured every 7200 s and only small ($< 3\%$) random variations were observed. Lorentz and polarization corrections were applied. An empirical absorption correction based on a series of ψ scans was applied to the data: $T_{\text{min}} = 0.863$, $T_{\text{max}} = 0.999$. The space group, $C2/c$, was determined by systematic absences (hkl , $h + k = \text{odd}$; $0k0$, $k = \text{odd}$; $h0l$, $l = \text{odd}$).

The structure was solved by heavy-atom methods using *SHELXS86* (Sheldrick, 1986) which revealed two independent positions of Mo. The remaining non-H atoms were located in successive difference Fourier syntheses. H atoms were not included in calculations. Atomic scattering factors including anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The structure was refined with full-matrix least squares using *Personal SDP* (B. A. Frenz & Associates, Inc., 1989) on a 80386-based IBM compatible PC. All atoms were refined anisotropically. $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 4F_o^2/[\sigma^2(I) + 0.01F_o^2]$. A secondary-extinction coefficient refined to a value of $\chi = 2.22 \times 10^{-7}$, where the correction factor $(1 + \chi I_c)^{-1}$ was applied to F_c . The final $R = 0.046$, $wR = 0.061$, $R_{\text{all}} = 0.059$ and $S = 3.137$ were obtained using 740 variables and 7459 observed

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Table 1. Positional parameters and their e.s.d.'s

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as:
 $(4/3)[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

	x	y	z	B _{eq} (Å ²)
Mo1	0.43781 (1)	0.31091 (5)	-0.01063 (3)	2.499 (9)
Mo2	0.18764 (1)	0.32159 (5)	0.22485 (3)	2.69 (1)
O01	0.41068 (9)	0.4057 (5)	-0.1644 (3)	5.8 (1)
O02	0.47567 (7)	0.3171 (5)	-0.1071 (3)	5.5 (1)
O03	0.15591 (7)	0.3699 (5)	0.0718 (3)	4.9 (1)
O04	0.22362 (7)	0.4011 (6)	0.1283 (3)	6.4 (1)
O51	0.4127 (1)	0.6340 (6)	0.1068 (3)	8.9 (2)
O52	0.40161 (7)	0.5719 (4)	-0.0111 (2)	4.0 (1)
O61	0.43937 (7)	0.6498 (5)	-0.0744 (3)	4.7 (1)
O62	0.47569 (7)	0.6164 (5)	-0.0520 (3)	5.4 (1)
O71	0.46532 (7)	0.2049 (5)	0.2074 (2)	4.4 (1)
O121	0.46752 (8)	0.0034 (6)	-0.0454 (3)	6.2 (1)
O122	0.46177 (7)	-0.0377 (5)	0.0724 (3)	5.0 (1)
O131	0.43855 (8)	0.1137 (6)	-0.1741 (3)	5.8 (1)
O132	0.40423 (8)	0.0457 (6)	-0.1810 (3)	6.7 (1)
O141	0.40017 (7)	0.2497 (5)	0.1543 (2)	5.1 (1)
O191	0.14887 (9)	0.0718 (6)	0.1397 (3)	7.6 (1)
O192	0.15449 (7)	-0.0219 (5)	0.2530 (3)	4.9 (1)
O201	0.21796 (8)	0.0519 (7)	0.0923 (3)	9.5 (2)
O202	0.18409 (8)	0.1120 (6)	0.0566 (3)	6.7 (1)
O211	0.21757 (8)	0.2101 (6)	0.4381 (3)	5.7 (1)
O261	0.18938 (7)	0.6515 (5)	0.1518 (3)	5.9 (1)
O262	0.15308 (8)	0.6585 (6)	0.1471 (3)	7.0 (2)
O271	0.22453 (8)	0.6003 (6)	0.2825 (3)	6.4 (1)
O272	0.21401 (8)	0.5926 (6)	0.3963 (3)	6.8 (1)
O281	0.15581 (7)	0.2246 (5)	0.4005 (3)	5.2 (1)
N1	0.46076 (7)	0.3440 (5)	0.1069 (3)	2.9 (1)
N2	0.40905 (7)	0.2080 (5)	0.0375 (3)	3.0 (1)
N3	0.21129 (8)	0.1868 (5)	0.3089 (3)	3.5 (1)
N4	0.16240 (7)	0.3615 (5)	0.3072 (3)	3.1 (1)
C01	0.4201 (1)	0.3715 (6)	-0.1065 (3)	3.4 (1)
C02	0.4622 (1)	0.3138 (6)	-0.0698 (3)	3.4 (1)
C03	0.16715 (9)	0.3500 (6)	0.1292 (3)	3.3 (1)
C04	0.2108 (1)	0.3724 (7)	0.1650 (4)	3.9 (1)
C1	0.47397 (9)	0.4523 (6)	0.0784 (3)	3.3 (1)
C2	0.4773 (1)	0.5416 (7)	0.1480 (4)	3.9 (1)
C3	0.4598 (1)	0.5289 (7)	0.1836 (4)	4.1 (2)
C4	0.44449 (9)	0.4285 (6)	0.1380 (3)	3.2 (1)
C5	0.43489 (9)	0.4841 (6)	0.0575 (3)	2.8 (1)
C51	0.4155 (1)	0.5725 (6)	0.0536 (3)	3.6 (1)
C52	0.3828 (1)	0.6597 (7)	-0.0205 (5)	5.2 (2)
C6	0.45374 (9)	0.5006 (6)	0.0201 (3)	2.8 (1)
C61	0.45758 (9)	0.5942 (6)	-0.0376 (3)	3.4 (1)
C62	0.4415 (1)	0.7324 (8)	-0.1390 (4)	6.1 (2)
C7	0.4722 (1)	0.2483 (6)	0.1564 (3)	3.5 (1)
C71	0.4942 (1)	0.2069 (7)	0.1401 (4)	4.4 (2)
C8	0.3989 (1)	0.1461 (6)	-0.0376 (3)	3.3 (1)
C9	0.3924 (1)	0.0147 (7)	0.0118 (4)	4.1 (2)
C10	0.4070 (1)	-0.0165 (7)	0.0513 (4)	4.3 (2)
C11	0.42350 (9)	0.0942 (6)	0.0672 (3)	3.2 (1)
C12	0.43732 (9)	0.1013 (6)	0.0017 (3)	3.0 (1)
C121	0.4568 (1)	0.0194 (6)	0.0041 (4)	3.8 (1)
C122	0.4818 (1)	-0.1200 (8)	0.0835 (5)	6.5 (2)
C13	0.4217 (1)	0.1355 (6)	-0.0660 (3)	3.2 (1)
C131	0.4207 (1)	0.0921 (6)	-0.1451 (4)	4.1 (2)
C132	0.4391 (2)	0.070 (1)	-0.2520 (4)	7.3 (2)
C14	0.39504 (9)	0.2591 (6)	0.0863 (4)	3.7 (1)
C141	0.3747 (1)	0.3324 (7)	0.0474 (4)	4.5 (2)
C15	0.2216 (1)	0.1231 (7)	0.2462 (3)	4.0 (2)
C16	0.2231 (1)	-0.0162 (8)	0.2734 (4)	4.9 (2)
C17	0.2061 (1)	-0.0385 (7)	0.3116 (4)	4.8 (2)
C18	0.1935 (1)	0.0881 (6)	0.3116 (3)	3.5 (1)
C19	0.18219 (9)	0.1141 (6)	0.2285 (3)	3.0 (1)
C191	0.1605 (1)	0.0536 (7)	0.1999 (4)	4.1 (2)
C192	0.1332 (1)	-0.0874 (8)	0.2350 (4)	5.5 (2)
C20	0.19999 (9)	0.1357 (6)	0.1858 (3)	3.3 (1)
C201	0.2014 (1)	0.0973 (7)	0.1071 (4)	4.1 (2)
C202	0.1846 (1)	0.0685 (9)	-0.0202 (4)	6.8 (2)
C21	0.2255 (1)	0.2202 (8)	0.3801 (4)	4.5 (2)
C211	0.2480 (1)	0.268 (1)	0.3749 (4)	6.3 (2)
C22	0.1791 (1)	0.4394 (7)	0.3626 (3)	3.7 (1)
C23	0.1639 (1)	0.5442 (7)	0.3865 (4)	4.6 (2)
C24	0.1477 (1)	0.5663 (7)	0.3261 (4)	4.7 (2)
C25	0.15165 (9)	0.4768 (6)	0.2618 (3)	3.5 (1)
C26	0.17354 (9)	0.5173 (6)	0.2353 (3)	3.2 (1)
C261	0.1712 (1)	0.6183 (7)	0.1754 (4)	4.0 (2)
C262	0.1874 (1)	0.7466 (9)	0.0893 (5)	7.5 (2)
C27	0.19098 (9)	0.4915 (6)	0.2991 (3)	3.3 (1)
C271	0.2114 (1)	0.5681 (7)	0.3215 (4)	4.3 (2)
C272	0.2344 (1)	0.6611 (9)	0.4308 (5)	7.4 (2)
C28	0.1483 (1)	0.2739 (7)	0.3395 (3)	3.9 (1)
C281	0.1259 (1)	0.2484 (8)	0.2938 (4)	4.9 (2)

Table 2. Selected bond distances (Å) and bond angles (°)

Numbers in parentheses are estimated e.s.d.'s in the least significant digits.

Mo1—N1	2.336 (3)	C2—C3	1.338 (5)
Mo1—N2	2.347 (3)	C3—C4	1.543 (5)
Mo1—C01	1.961 (3)	C4—C5	1.563 (4)
Mo1—C02	1.969 (3)	C5—C6	1.437 (4)
Mo1—C5	2.198 (4)	C5—C51	1.496 (5)
Mo1—C6	2.234 (4)	C6—C61	1.463 (5)
Mo1—C12	2.199 (3)	C1—C6	1.559 (5)
Mo1—C13	2.228 (3)	C8—C9	1.520 (5)
O01—C01	1.147 (3)	N1—C1	1.527 (5)
C8—C13	1.566 (4)	O02—C02	1.141 (4)
N1—C4	1.504 (4)	C9—C10	1.348 (4)
N1—C7	1.433 (5)	C10—C10	1.532 (4)
N2—C8	1.516 (4)	C11—C12	1.553 (4)
N2—C11	1.522 (5)	C12—C13	1.448 (4)
N2—C14	1.422 (5)	C12—C121	1.465 (5)
C1—C2	1.533 (5)	C13—C131	1.468 (4)
Mo2—N4	2.339 (6)	C17—C18	1.530 (5)
Mo2—C03	1.955 (5)	C18—C19	1.545 (4)
Mo2—C04	1.982 (5)	C19—C20	1.447 (5)
Mo2—C19	2.193 (5)	C19—C191	1.484 (5)
Mo2—C20	2.234 (5)	C20—C201	1.470 (5)
Mo2—C26	2.238 (7)	C15—C20	1.565 (5)
Mo2—C27	2.198 (5)	C22—C23	1.542 (5)
O03—C03	1.148 (5)	N3—C15	1.523 (5)
C22—C27	1.542 (5)	O04—C04	1.145 (6)
N3—C18	1.506 (6)	C23—C24	1.352 (6)
N3—C21	1.448 (6)	C24—C25	1.525 (6)
N4—C22	1.526 (6)	C25—C26	1.555 (5)
N4—C25	1.533 (7)	C26—C27	1.445 (6)
N4—C28	1.443 (7)	C26—C261	1.487 (6)
C15—C16	1.530 (6)	C27—C271	1.485 (6)
N1—Mo1—N2	96.8 (1)	C5—Mo1—C6	37.8 (1)
N1—Mo1—C01	152.7 (1)	C5—Mo1—C12	139.4 (1)
N1—Mo1—C02	94.4 (1)	C5—Mo1—C13	147.8 (1)
N1—Mo1—C5	58.7 (1)	C6—Mo1—C12	150.5 (1)
N1—Mo1—C6	58.6 (1)	C6—Mo1—C13	168.2 (1)
N1—Mo1—C12	94.3 (1)	C12—Mo1—C13	38.2 (1)
N1—Mo1—C13	132.1 (1)	N2—Mo1—C01	97.0 (1)
N2—Mo1—C02	152.6 (1)	N2—Mo1—C5	92.3 (1)
N2—Mo1—C6	129.9 (1)	N2—Mo1—C12	59.0 (1)
N2—Mo1—C13	58.6 (1)	C01—Mo1—C02	83.8 (1)
C01—Mo1—C5	97.3 (1)	C01—Mo1—C6	94.6 (1)
C01—Mo1—C12	113.1 (1)	C01—Mo1—C13	75.1 (1)
C02—Mo1—C5	114.9 (1)	C02—Mo1—C6	77.0 (1)
C02—Mo1—C12	95.3 (1)	C02—Mo1—C13	95.7 (1)
N3—Mo2—N4	96.5 (2)	C19—Mo2—C20	38.1 (1)
N3—Mo2—C03	150.9 (2)	C19—Mo2—C26	146.7 (2)
N3—Mo2—C04	94.5 (2)	C19—Mo2—C27	141.1 (2)
N3—Mo2—C19	58.3 (2)	C20—Mo2—C26	166.9 (2)
N3—Mo2—C20	58.3 (2)	C20—Mo2—C27	152.1 (2)
N3—Mo2—C26	134.1 (2)	C26—Mo2—C27	38.0 (2)
N3—Mo2—C27	96.7 (2)	N4—Mo2—C03	96.8 (2)
N4—Mo2—C04	153.6 (3)	N4—Mo2—C19	92.1 (2)
N4—Mo2—C20	130.0 (2)	N4—Mo2—C26	58.6 (2)
N4—Mo2—C27	59.1 (2)	C03—Mo2—C04	84.7 (2)
C03—Mo2—C19	95.5 (2)	C03—Mo2—C20	93.7 (2)
C03—Mo2—C26	74.5 (2)	C03—Mo2—C27	112.3 (2)
C04—Mo2—C19	114.0 (2)	C04—Mo2—C20	75.9 (2)
C04—Mo2—C26	97.0 (2)	C04—Mo2—C27	95.9 (2)

reflections [$I > 3\sigma(I)$]. The largest shift/e.s.d. was 0.06; maximum and minimum residual electron densities in the final difference Fourier map were 0.67 and $-0.42 e \text{ \AA}^{-3}$.

Final positional parameters are presented in Table 1.* Selected bond lengths and angles are listed in Table 2. Fig. 1 shows the ORTEP drawing (Johnson,

* Lists of structure factors, anisotropic displacement parameters, and a complete list of bond distances, bond angles and torsion angles for the two independent molecules have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54364 (70 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

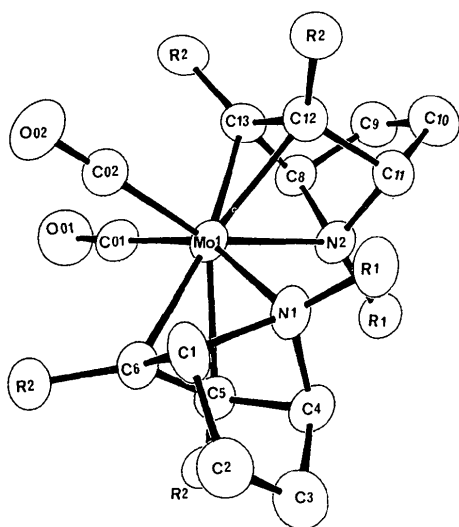


Fig. 1. ORTEP drawing of the title compound with 50% probability ellipsoids. $R_1 = \text{COCH}_3$, $R_2 = \text{COOCH}_3$.

1970; B. A. Frenz & Associates, Inc., 1989) of the molecule and the atomic labeling scheme.

Related literature. The crystal structure of a 7-azanorbornadiene derivative-Fe complex compound has been reported by Sun, Chow & Liu (1990).

These studies are supported by the National Science Council (NSC80-0208-M031-05) of Taiwan.

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Acta Cryst. (1991). **C47**, 2461-2463

Structure of Potassium 2-Pyridonide Monohydrate

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(Received 14 January 1991; accepted 11 June 1991)

Abstract. $\text{K}^+ \cdot \text{C}_5\text{H}_4\text{NO}^- \cdot \text{H}_2\text{O}$, $M_r = 151.20$, orthorhombic, $Pbcn$, $a = 6.2727$ (21), $b = 7.0732$ (10), $c = 28.502$ (10) Å, $V = 1265$ Å³, $Z = 8$, $D_x = 1.588$ Mg m⁻³, $\bar{\lambda}(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.752$ mm⁻¹, $F(000) = 624$, $T = 120.0$ (1) K, $R = 0.0274$ for 910 unique observed reflections. The structure comprises hydrogen-bonded planes of K^+ ions and water molecules to which the pyridonide anions are hydrogen-bonded orthogonally on each side, resulting in a structure which has alternating hydrophilic and hydrophobic zones.

Experimental. Title compound prepared by reaction of aqueous solutions of KOH and 2-pyridone, crystals obtained by recrystallization from *n*-propanol/diethyl ether. Colourless plate, $0.12 \times 0.35 \times 0.74$ mm, Stoe STADI-4 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986), cell parameters from 2θ values of 21

reflections measured at $\pm\omega$ ($30 < 2\theta < 32^\circ$). For data collection at $T = 120$ K, ω scans with scan width $(1.32 + 0.35 \tan \theta)^\circ$, $2\theta_{\text{max}} = 50^\circ$, $h 0 \rightarrow 7$, $k 0 \rightarrow 8$, $l 0 \rightarrow 33$, no significant crystal movement or decay, no absorption correction, 1369 unique reflections, giving 910 with $F > 4\sigma(F)$. Structure solution from a Patterson synthesis (K) followed by iterative cycles of least-squares refinement and difference Fourier synthesis, and refinement using full-matrix least-squares on F (*SHELX76*; Sheldrick, 1976). Anisotropic thermal parameters for all non-H atoms, H atoms refined freely with individual isotropic thermal parameters, secondary-extinction parameter refined to $3.7(12) \times 10^{-7}$. At final convergence, $R = 0.0274$, $wR = 0.0475$, $S = 1.339$ for 107 parameters, $(\Delta/\sigma)_{\text{max}}$ in final cycle 0.04, max. and min. $\Delta\rho$ in final ΔF synthesis 0.27, -0.31 e Å⁻³ respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0.00415F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (*SHELX76*; Sheldrick, 1976) except for K (Cromer & Mann, 1968). Atomic coordinates and equivalent isotropic thermal parameters are given in

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