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A New Mixed-Ligand Complex of the Quadruply Bonded Ditungsten Unit. Bis(*N*,*N'*-diphenylacetamidinato)bis(2,4-dimethyl-6-hydroxypyrimidinato)ditungsten Bis(tetrahydrofuranate)

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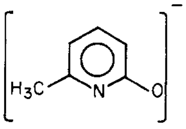
Reaction of $W_2(dmhp)_4$ (dmhp = the anion of 2,4-dimethyl-6-hydroxypyrimidine) with the lithium salt of *N*,*N'*-diphenylacetamidine, $Li[(PhN)_2CCH_3]$, has given a product in which two dmhp ligands have been replaced, in transoid fashion, by two $[(PhN)_2CCH_3]$ ligands. This red crystalline product, $W_2(dmhp)_2[(PhN)_2CCH_3]_2\cdot 2THF$, is air sensitive but thermally stable. It is only sparingly soluble in several common solvents and insufficiently volatile to give a mass spectrum under the usual conditions. It crystallizes in space group $P2_1/n$ with $Z = 2$, thus requiring the molecules to lie on crystallographic inversion centers. The W–W distance 2.174 (1) Å is very similar to that in $W_2(dmhp)_4$. The unit cell dimensions are $a = 11.399$ (3) Å, $b = 10.431$ (1) Å, $c = 20.002$ (4) Å, $\beta = 101.9$ (3)°, and $V = 2327.4$ (1.5) $Å^3$. The structure has been refined to $R_1 = 0.039$ and $R_2 = 0.053$ by using anisotropic thermal parameters for all atoms except those in the THF molecule.

Introduction

Compounds containing quadruple bonds between two tungsten atoms are still quite rare. Aside from $W_2(C_8H_8)_3$[1] which is rather different from all the others (an 18-electron case as against the formal 16-electron structures of the others), there are only $[W_2(CH_3)_8]^{4-}$ and the closely related $[W_2Cl_n(CH_3)_{8-n}]^{4-}$ ions,[2] $W_2(mhp)_4$, which is by far the most stable,[3] $W_2(map)_4$,[4] and $W_2(dmhp)_4$,[5] where mhp stands for the anion of 6-methyl-2-hydroxypyridine (**1**), map stands for the anion of 6-methyl-2-aminopyridine (**2**), and dmhp represents the anion of 2,4-dimethyl-6-hydroxypyrimidine (**3**). In contrast to the ease of preparation and stability of $Mo_2(O_2CR)_4$ and $Re_2(O_2CR)_4X_2$ compounds, no $W_2(O_2CR)_4$ compound has yet been made.



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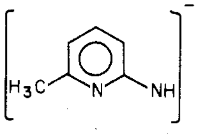

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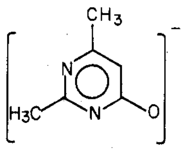

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We have been attempting to reach this goal both directly and indirectly. One indirect approach is to prepare W_2 compounds with carboxylate-like ligands such as amidinates, $RNCR'NR^-$. We have examined several reactions in which amidinate ligands are allowed to react with $W_2(mhp)_4$, $W_2(map)_4$, or $W_2(dmhp)_4$ compounds with the object of obtaining partial or complete replacement of the original ligands by the amidinate ligands. We report here the result of one such effort in which $[PhNC(CH_3)NPh]^-$ ions replace two of the four $dmhp^-$ ions in $W_2(dmhp)_4$ to give $W_2(dmhp)_2[(PhN)_2CCH_3]$, which forms crystals containing THF of solvation.

Experimental Section

N,*N'*-Diphenylacetamidine, $C_6H_5N(H)C(CH_3)NC_6H_5$, was prepared by heating acetanilide and phosphorus trichloride.[6] Recrystallization from ethanol afforded almost colorless needles, mp 130 °C. $W_2(dmhp)_4\cdot 0.5(diglyme)$ was made according to the procedure described earlier.[5]

Bis(*N*,*N'*-diphenylacetamidinato)bis(2,4-dimethyl-6-hydroxypyrimidinato)ditungsten. A solution of (*N*,*N'*-diphenylacetamidinato)lithium was prepared by adding *n*-butyllithium in hexane to 0.21 g (1 mM) of $C_6H_5N(H)C(CH_3)NC_6H_5$ in 25 mL of THF. Complete conversion of the amidine to its colorless anion was indicated by the yellow color which developed with more than 1 equiv of the base, *n*-BuLi, probably due to formation of a dianion. To this solution, 0.23 g (0.25 mM) of $W_2(dmhp)_4\cdot 0.5(diglyme)$ was added, and the mixture was stirred at room temperature overnight.

After 16 h a red precipitate had formed. The remaining solvent was removed by filtration, and some of the air-sensitive material was dissolved in THF. A layer of hexane was carefully placed on top of the dark red solution, and several days later slow diffusion had produced deep red crystals which were collected for X-ray crystallography and were recognized as the title compound. Since further substitution of the dmhp ligand by the amidinato group was not achieved at room temperature, the reaction was repeated in 10 mL of refluxing 2-methoxyethyl ether (diglyme; bp 162 °C). After 3 h of boiling the mixture, the red precipitate was collected by filtration and dissolved in THF; however, the crystals growing at the THF–hexane interface proved to be again the twofold substituted species as established by X-ray diffraction. A mass spectrum of this compound could not be obtained, probably because of low volatility.

X-ray Data Collection. Crystals of $W_2[(PhN)_2CCH_3]_2(dmhp)_2\cdot 2THF$ were transferred, in a nitrogen filled drybox, to a Petri dish containing degassed mineral oil. The crystals were then removed from the drybox and examined under a microscope. A crystal suitable for X-ray analysis was chosen and transferred to a slide on which there was a drop of soft epoxy cement. The crystal was then coated with the epoxy cement and secured to the inside of a glass capillary, sealed under argon and mounted on a Syntex $P\bar{1}$ automatic diffractometer.

Preliminary rotation and oscillation photographs indicated that the crystal was monoclinic, and examination of selected reflections showed that it was a single crystal of good quality. ω scans showed that several intense reflections had widths at half-height of less than 0.3°. A preliminary data set showed absences for $0k0$, $k = 2n + 1$, and $h0l$, $h + l = 2n + 1$, indicating the space group $P2_1/n$. Careful centering of 15 reflections in the range $23 < 2\theta < 32°$, selected to give varied crystal orientations, gave unit cell parameters $a = 11.399$ (3) Å, $b = 10.431$ (1) Å, $c = 20.002$ (4) Å, $\beta = 101.9$ (3)°, and $V = 2327.4$ (1.5) $Å^3$. This volume is consistent with $Z = 2$. The procedures preliminary to data collection have been described previously.[7]

All data were collected by using Mo Kα (λ 0.71073 Å) radiation monochromatized in the incident beam with a graphite crystal. A total of 2723 independent reflections with $0 < 2\theta \leq 45°$ were collected at 22 ± 4 °C by using the θ–2θ scan technique. A variable scan rate from 4 to 24° min^{-1} was used with a scan to background time ratio of 2. The intensities of three standard reflections were measured after every 97 reflections and showed no significant variation during the period of data collection. Lorenz and polarization corrections were applied.[8] An empirical absorption correction ($\mu = 52.695$ cm^{-1}), based on ψ scans at $\chi = 90°$, was made to the data. A total of seven curves were used covering the range $7 < 2\theta < 32°$. Each reference curve was collected in 10° intervals from $0 \leq 2\theta \leq 360°$. The minimum and maximum correction factors were 0.666 and 1.000, respectively.

Solution and Refinement. The structure was solved[8] in the monoclinic space group $P2_1/n$. The position of the one crystallographically independent tungsten atom was found by solution of the three-dimensional Patterson function. Subsequent Fourier and difference syntheses gave the positions of the remaining nonhydrogen atoms. Full-matrix anisotropic least-squares refinement of all atomic coordinates and temperature factors, except those of the THF molecule

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Table I. Positional and Thermal Parameters and Their Estimated Standard Deviations

atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
W(1)	0.50821 (4)	0.4973 (1)	0.55511 (2)	2.32 (2)	2.92 (2)	1.65 (2)	0.10 (4)	0.33 (1)	0.11 (5)
O(1)	0.4333 (9)	0.313 (1)	0.4326 (5)	4.2 (4)	2.5 (4)	2.2 (4)	−0.1 (4)	0.6 (4)	−0.4 (4)
N(1)	0.4494 (10)	0.299 (1)	0.5477 (7)	2.0 (4)	3.0 (6)	3.4 (6)	−0.2 (5)	0.0 (4)	0.4 (5)
N(2)	0.3974 (11)	0.110 (1)	0.5995 (7)	3.6 (5)	3.6 (7)	4.5 (7)	0.1 (5)	1.3 (5)	1.0 (6)
N(3)	0.3118 (9)	0.561 (1)	0.4320 (7)	1.8 (4)	3.7 (5)	3.5 (6)	0.2 (4)	0.5 (4)	−2.2 (5)
N(4)	0.3273 (9)	0.551 (1)	0.5482 (6)	2.5 (4)	2.8 (5)	1.5 (4)	−0.2 (4)	0.5 (4)	−1.4 (4)
C(1)	0.422 (1)	0.246 (1)	0.4836 (8)	3.6 (6)	2.4 (6)	2.4 (6)	1.1 (6)	0.9 (5)	0.5 (6)
C(2)	0.437 (1)	0.226 (1)	0.6032 (8)	3.2 (6)	2.5 (6)	4.1 (7)	0.8 (5)	1.2 (5)	2.4 (6)
C(3)	0.469 (2)	0.289 (2)	0.6735 (8)	5.8 (8)	4.7 (9)	1.5 (6)	0.4 (7)	0.8 (6)	−1.2 (7)
C(4)	0.380 (1)	0.120 (2)	0.4774 (9)	3.8 (7)	3.4 (8)	3.4 (8)	−0.3 (7)	0.5 (6)	−0.7 (7)
C(5)	0.367 (1)	0.048 (2)	0.5351 (11)	4.0 (7)	1.7 (6)	8 (1)	0.4 (5)	1.5 (7)	0.9 (7)
C(6)	0.324 (2)	−0.088 (2)	0.5334 (11)	9 (1)	1.7 (7)	6 (1)	−2.6 (7)	2.5 (8)	−0.3 (7)
C(7)	0.257 (1)	0.575 (1)	0.4835 (9)	2.8 (5)	2.1 (6)	4.4 (8)	0.3 (5)	1.3 (5)	0.7 (6)
C(8)	0.124 (1)	0.609 (2)	0.4738 (9)	2.7 (6)	5.0 (9)	3.4 (7)	1.4 (6)	0.8 (5)	1.5 (7)
C(9)	0.276 (1)	0.548 (2)	0.6060 (8)	1.7 (5)	5.4 (9)	3.8 (7)	0.8 (5)	0.9 (5)	0.2 (7)
C(10)	0.321 (2)	0.632 (2)	0.6596 (9)	5.7 (8)	4.5 (9)	2.7 (7)	0.7 (8)	0.4 (7)	−1.3 (7)
C(11)	0.271 (2)	0.619 (2)	0.7202 (10)	7 (1)	7 (1)	3.3 (8)	0 (1)	1.6 (7)	−0.2 (9)
C(12)	0.190 (2)	0.528 (2)	0.7282 (9)	4.5 (7)	10 (2)	4.3 (8)	1.2 (9)	1.4 (6)	3.5 (9)
C(13)	0.147 (1)	0.442 (2)	0.6735 (11)	2.0 (6)	6 (1)	6 (1)	0.7 (7)	−0.2 (6)	2.3 (9)
C(14)	0.194 (1)	0.450 (2)	0.6118 (8)	2.9 (6)	5.4 (9)	2.3 (6)	0.3 (6)	0.3 (5)	2.2 (6)
C(15)	0.246 (1)	0.591 (1)	0.3622 (7)	2.6 (5)	2.9 (7)	1.5 (6)	−0.4 (5)	−0.6 (5)	−0.4 (6)
C(16)	0.210 (1)	0.715 (2)	0.3429 (9)	3.8 (7)	4.4 (8)	3.3 (8)	1.2 (7)	0.2 (6)	1.4 (7)
C(17)	0.146 (1)	0.742 (2)	0.2770 (9)	4.0 (7)	5 (1)	3.5 (8)	0.7 (7)	0.2 (7)	1.4 (8)
C(18)	0.121 (1)	0.640 (2)	0.2294 (9)	3.1 (7)	10 (1)	2.7 (8)	1.5 (9)	−0.2 (6)	2 (1)
C(19)	0.159 (2)	0.514 (2)	0.2481 (8)	6.4 (8)	7 (1)	2.7 (6)	2.6 (9)	−1.1 (6)	−3.6 (8)

atom	x	y	z	B, Å^2	atom	x	y	z	B, Å^2
C(20)	0.220 (1)	0.492 (2)	0.3144 (8)	4.3 (3)	C(23)	0.389 (4)	0.659 (5)	0.1474 (25)	18.2 (16)
C(21)	0.430 (5)	0.517 (5)	0.1400 (28)	20.1 (19)	C(24)	0.040 (4)	0.236 (5)	0.3969 (25)	18.1 (17)
C(22)	0.553 (5)	0.571 (6)	0.1240 (32)	23.5 (22)	C(25)	0.474 (4)	0.639 (6)	0.0596 (25)	20.0 (19)

[a] The form of the anisotropic thermal parameter is $\exp[-\frac{1}{4}(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

Table II. Bond Distances (Å)

W(1)-W(1)	2.174 (1)	C(7)-C(8)	1.53 (2)
O(1)′	2.09 (1)	C(9)-C(10)	1.40 (2)
N(1)	2.17 (1)	C(14)	1.41 (2)
N(3)′	2.11 (1)	C(10)-C(11)	1.45 (2)
N(4)	2.11 (1)	C(11)-C(12)	1.36 (3)
O(1)-C(1)	1.26 (2)	C(12)-C(13)	1.43 (3)
N(1)-C(1)	1.37 (2)	C(13)-C(14)	1.44 (2)
C(2)	1.37 (2)	C(15)-C(16)	1.38 (2)
N(2)-C(2)	1.30 (2)	C(20)	1.40 (2)
C(5)	1.42 (2)	C(16)-C(17)	1.40 (2)
N(3)-C(7)	1.32 (2)	C(17)-C(18)	1.42 (3)
C(15)	1.48 (2)	C(18)-C(19)	1.41 (3)
N(4)-C(7)	1.40 (2)	C(19)-C(20)	1.38 (2)
C(9)	1.40 (2)	C(21)-C(22)	1.60 (7)
C(2)-C(3)	1.53 (2)	C(23)	1.56 (6)
C(4)-C(1)	1.39 (2)	C(22)-C(25)	1.58 (7)
C(5)	1.41 (3)	C(23)-C(24)	1.54 (6)
C(5)-C(6)	1.50 (2)	C(24)-C(25)	1.36 (7)

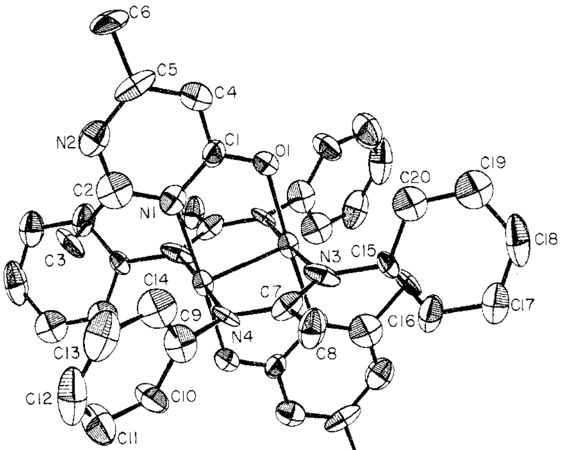

Figure 1. An ORTEP drawing of the $W_2(dmhp)_2(PhNCMeNPh)_2$ molecule, showing the atom numbering scheme.

Table III. Bond Angles (deg)

W(1)′-W(1)-O(1)′	93.0 (3)	C(1)-C(4)-C(5)	121 (1.7)
N(1)	89.7 (4)	N(2)-C(5)-C(4)	117 (1.6)
N(3)	90.6 (4)	C(6)	118 (2)
N(4)	92.7 (3)	C(4)-C(5)-C(6)	125 (2)
O(1)′-W(1)-N(1)	177.2 (4)	N(3)-C(7)-N(4)	115 (1)
N(3)′	88.8 (4)	C(8)	123 (1.4)
N(4)	92.3 (4)	N(4)-C(7)-C(8)	122 (1.3)
N(1)-W(1)-N(3)′	90.7 (5)	N(4)-C(9)-C(10)	118 (1.5)
N(4)	87.9 (4)	C(14)	119 (1.5)
N(3)′-W(1)-N(4)	176.4 (5)	C(10)-C(9)-C(14)	123 (1.6)
W(1)′-O(1)-C(1)	121 (1)	C(9)-C(10)-C(11)	116 (1.7)
N(1)-C(1)	117 (1)	C(10)-C(11)-C(12)	124 (2)
C(2)	123 (1)	C(11)-C(12)-C(13)	119 (2)
C(1)-N(1)-C(2)	119 (1)	C(12)-C(13)-C(14)	120 (1.6)
C(2)-N(2)-C(5)	120 (2)	C(9)-C(14)-C(13)	119 (1.6)
W(1)′-N(3)-C(7)	123 (1)	N(3)-C(15)-C(16)	122 (1.3)
C(15)	118 (1)	C(20)	119 (1.3)
C(7)-N(3)-C(15)	119 (1)	C(16)-C(15)-C(20)	119 (1.5)
W(1)-N(4)-C(7)	118 (1)	C(15)-C(16)-C(17)	121 (1.7)
C(9)	121 (1)	C(16)-C(17)-C(18)	118 (1.7)
C(7)-N(4)-C(9)	121 (1)	C(17)-C(18)-C(19)	121 (1.5)
O(1)-C(1)-N(1)	119 (1)	C(18)-C(19)-C(20)	118 (2)
C(4)	123 (1.5)	C(15)-C(20)-C(19)	122 (2)
N(1)-C(1)-C(4)	118 (1.4)	C(22)-C(21)-C(23)	89 (5)
N(1)-C(2)-N(2)	124 (1.7)	C(21)-C(22)-C(25)	87 (4)
C(3)	118 (1.4)	C(21)-C(23)-C(24)	103 (5)
N(2)-C(2)-C(3)	118 (1.5)	C(23)-C(24)-C(25)	97 (5)
		C(22)-C(25)-C(24)	86 (5)

and one of the phenyl carbons which continued to have a nonpositive definite thermal ellipsoid when refined anisotropically, gave final discrepancy factors of

$$R_1 = (\sum||F_o| - |F_c||)/\sum|F_o| = 0.039$$

$$R_2 = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2} = 0.053$$

and a goodness of fit parameter of 1.272. The function $\sum w(|F_o| - |F_c|)^{1/2}$ was minimized with the weighting factor, w, equal to $4F_o^2/\sigma(F_o^2)^2$. All structure factor calculations and least-squares refinements were executed by using only those 1624 reflections for which $F_o^2 > 3\sigma(F_o)^2$. Atomic scattering factors were those of Cromer and Waber.[9]

Table IV. Some Planes and Dihedral Angles, Deg

Planes[a]	displacements	error
P_1: N(4)-W(1)-W(1)′-N(3)		
$A = -0.2708$	N(4) = 0.012	0.012
$B = -0.9601$	W(1) = −0.013	0.001
$C = -0.0702$	W(1)′ = −0.013	0.000
$D = -6.6815$	N(3) = −0.012	0.013
P_2: C(15)-C(16)-C(17)-C(18)-C(19)-C(20)		
$A = 0.9435$	C(15) = −0.004	0.014
$B = 0.1664$	C(16) = 0.009	0.016
$C = -0.2865$	C(17) = −0.006	0.017
$D = 0.2394$	C(18) = −0.001	0.017
	C(19) = 0.005	0.018
	C(20) = −0.003	0.016
P_3: C(9)-C(10)-C(11)-C(12)-C(13)-C(14)		
$A = -0.6772$	C(9) = 0.024	0.016
$B = 0.6326$	C(10) = −0.019	0.019
$C = -0.3758$	C(11) = 0.007	0.021
$D = -1.3091$	C(12) = −0.001	0.020
	C(13) = 0.005	0.019
	C(14) = −0.017	0.016

Dihedral Angles (deg) between Planes

P_1/P_2	−66.7	P_1/P_3	−66.6	P_2/P_3	−64.8

[a] Each plane, P_n, is defined by $A_nX + B_nY = C_nZ = D_n$.

Anomalous dispersion effects were included in the scattering factors of tungsten. The observed and final calculated structure factors are available as supplementary material.

Results and Discussion

The structure of the molecule is shown in Figure 1, which also defines the atom numbering scheme. The atomic positional and thermal parameters are listed in Table I. Bond distances and angles are given in Tables II and III, respectively. The equations for some planes and the dihedral angles between them are given in Table IV.

The molecules reside on crystallographic sites of inversion symmetry. The symmetry approximates to C_{2h} except for the twist angles of the phenyl groups which are inconsistent with this.

The THF molecules are not coordinated to the tungsten atoms but are present simply as solvent of crystallization, filling voids between the molecules. They reside on the fourfold general position. Unfortunately, they failed to refine satisfactorily despite persistent efforts to accomplish this. We have been unable to establish whether this is the result of disorder, partial occupation, large thermal motions, or some combination of two or all three of these. Since we could not distinguish the oxygen atom from the carbon atoms, we refined the THF molecule as five carbon atoms, C(21)–C(25) in Table I. On this basis we obtained C–C distances ranging from 1.36 (7) to 1.60 (7) Å and angles ranging from 87 (4)° to 103 (5)° with isotropic thermal parameters from 18.1 to 23.5 Å^2.

Returning now to the $W_2(dmhp)_2(PhNCMeNPh)_2$ molecule, we note that it has like ligands trans to each other and that the dmhp ligands are arranged so that the O and N atoms are trans on each tungsten atom. The W–W distance, 2.174 (1) Å, is similar to those found in other isoelectronic compounds containing W–W quadruple bonds. It is in fact slightly longer than all of these others, viz., 2.164 (1) Å in $W_2(map)_4$,[4] 2.161 (1) Å in $W_2(mhp)_4$,[3] 2.155 (2) Å in $W_2(dmhp)_4 \cdot {}^1/_2$(diglyme),[5] and 2.163 (1) Å in $W_2(dmhp)_4 \cdot$diglyme.[5] The O trans to N arrangement of the dmhp ligands differs from the N trans to N and O trans to O arrangement found in $W_2(dmhp)_4$, but it should be noted that only the observed arrangement would maintain the equivalence of the metal atoms in the present case. It may be noted that the $Mo_2(DMP)_4$ (DMP = 2,6-dimethoxyphenyl) molecule has different atoms, C and O, trans to each other.[10]

The W–O and W–N distances to the dmhp ligands are 2.09 (1) and 2.17 (1) Å, respectively. These distances agree well with the distance observed in $W_2(dmhp)_4 \cdot$diglyme[5] [W–O = 2.064 (3) (av); W–N = 2.143 (4) Å (av)]. The C–C and C–N distances within the dmhp ligands show no unusual features and do not differ significantly from those observed in $W_2(dmhp)_4$.

The $PhNC(CH_3)NPh$ ligands are symmetrically bonded to the tungsten atoms with W–N distances of 2.11 (1) Å. The C–N distances within the ligands range from 1.32 (2) to 1.48 (2) Å. This large range reflects both the inaccuracy of the data and the difficulties inherent in accurately locating light atom positions in the presence of an atom as large as tungsten.

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Registry No. $W_2[(PhN)_2CCH_3]_2(dmhp)_2 \cdot 2THF$, 71630-66-5; $W_2(dmhp)_4 \cdot 0.5$(diglyme), 69351-89-9; $C_6H_5N(H)C(CH_3)NC_6H_5$, 621-09-0.

Supplementary Material Available: A table of final calculated and observed structure factors (7 pages). Ordering information is given on any current masthead page.

References and Notes

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