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# The Structure of Iodo-N-cyclohexyldiazenidobis[1,2-bis(diphenylphosphino)ethane] molybdenum. A Re-investigation

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In a previous paper, we [1] were able to show that the cyclohexyldiazenido group in $MoI(NNC_6H_{11})(dppe)_2$ (*1*) (where dppe = 1,2-bis(diphenylphosphino)ethane) was bonded in a singly-bent fashion to the molybdenum atom. In that article we also noted that the single crystals initially obtained for the benzene solvate of (*1*) were not of the highest quality and the unusually short N–N distance determined crystallographically was probably an artifact of the diffraction data which resulted from this poor crystal quality. We have since been able to obtain much better crystals of the benzene solvate of (*1*) and it has been structurally characterized by X-ray diffraction techniques [2]. Three-dimensional diffraction data on these crystals of (*1*) were collected on a computer-controlled four-circle Syntex $P\bar{1}$ Autodiffractometer using graphite monochromated $MoK\bar{\alpha}$ radiation and full (1° wide) $\omega$ scans. Structural parameters have been refined to convergence in cycles of empirically-weighted full-matrix least-squares refinement which employed variable positional and anisotropic thermal parameters for all nonhydrogen atoms and fixed parameters for idealized isotropic hydrogen atoms.

Crystal and refinement data are as follows:

$MoI(NNC_6H_{11})(P_2C_{26}H_{24})_2 \cdot 0.5C_6H_6$: monoclinic, $P2_1/c$; $a = 12.661(5)$, $b = 21.286(6)$, $c = 20.395(4)$ Å, $\beta = 99.07(3)$, $Z = 4$; $d_{calcd} = 1.428$ g cm$^{-3}$, $d_{measd} = 1.40$ g cm$^{-3}$; $R$ (unweighted, based on F) = 0.053 for 3701 independent reflections having $2\theta_{MoK\alpha} < 55°$ and $I > 3\sigma(I)$ (Fig. 1).

Bond lengths and bond angles around the molybdenum atom and within the alkyldiazenido ligand from this refinement are: Mo–$N_1$, 1.834(9); $N_1$–$N_2$, 1.155(12); $N_2$–C, 1.46(3); Mo–I, 2.884(2); Mo–P, 2.513(3,4,8,4) Å; Mo–$N_1$–$N_2$ 177(1); and $N_1$–$N_2$–C 132(1)° **. The previous values for the Mo–N, N–N and N–C bond lengths and the N–N–C bond angle were 1.95(1), 0.91(1), 1.39(4) Å and 142(2)°, respectively. The present N–N bond length is much more reasonable and slightly (0.039 Å) longer than the 1.118(8) Å N–N separation found in the parent bis(dinitrogen) complex, $Mo(N_2)_2(dppe)_2$ [3] but is significantly (*ca.* 0.10 Å) shorter than a normal organic N=N double bond; *e.g.*, 1.243(6) Å in *trans*-$C_6H_5N_2C_6H_5$ [6]. These most recent parameters are in good agreement with the qualitative molecular orbital picture of the structure of diazenido complexes as presented by Dubois and Hoffmann [5].

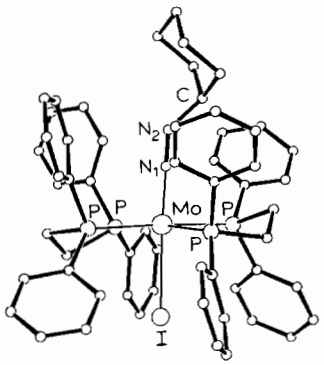


Fig. 1. Structural representation based on crystal data.

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## References

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**The first number in parentheses is the root mean square estimated standard deviation of an individual datum. The second and third numbers, when given are the average and maximum deviations from the average value, respectively. The forth number represents the number of individual measurements which are included in the average value.