This Week's Citation Classic

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Corfield P W R, Doedens R J & Ibers J A. Studies of metal-mtrogen multiple bonds I The crystal and molecular structure of nitridodichlorotris(diethylphenyl-phosphine)rhenium(V), $ReNCl_2[P(C_2H_5)_2C_6H_5]_3$. *Inorg. Chem.* **6**:197-204, 1967. [Dept. Chemistry, Northwestern Univ., Evanston, IL]

With the determination of the crystal structure of Re $NCl_2[P(C_2H_5)_2C_6H_5]_3$ as an illustration, procedures for the collection of reliable X-ray diffraction data and for the solution and refinement of structures of this type are described. [The *SCI®* indicates that this paper has been cited over 760 times since 1967.]

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"In the Spring of 1965 I moved to Northwestern University from Brook-haven National Laboratory. Shortly after my arrival I took delivery of a Picker card-programmed automatic dif-fractometer, number six in their series. This device represented a great advance in four-circle diffractometers for the collection of X-ray diffraction data from single crystals. Such data are the basis of crystal structure determinations. I had had experience with a four-circle diffractometer for neutron diffraction at the Graphite Reactor at Brookhaven, but alignment and use of that diffractometer proved to be very different from that of the X-ray diffractometer. Although the Picker instrument came with alignment instructions, these were minimal, and I quickly realized that alignment and utilization of the instrument for the efficient collection of accurate diffraction data would have to be worked out at North-western. That fall, two postdoctorals, Peter Corfield from Pittsburgh and Bob Doedens from Wisconsin, arrived and the three of us continued the self-education process on diffractometry that I had begun. Peter, Bob, and I worked on the various problems into early 1966. We devised a practical alignment procedure and we arrived at an understanding of the many factors which are essential for the collection of a reliable set of X-ray diffraction data, especially from an absorbing crystal. In addition, we adapted and wrote a number of computer programs for the CDC 3400 computer. These programs were necessary for the efficient operation of the diffractometer and for the facile solution and refinement of crystal structures.

"In the context of a structure determination of a rhenium nitrido complex, we discussed in some detail our procedures for data collection and for structure solution and refinement in our paper, which was submitted in August of 1966. Some 13 years later I find that with few exceptions, such as the acquisition of data at low temperatures,[1] the procedures we devised I still follow. Although the molecular structure reported in our paper is an interesting and important one, it is one of many these days. Presumably, the frequent citation of our paper arises because others found our discussion of the experimental methods of data collection and the details of structure refinement of value. The fact that the paper was published in *Inorganic Chemistry,* a journal where few crystal structure determinations were published in 1967 but where a great many are now, may also account for its frequent citation."

1. **Huffman J C**. *A crystallographic study of electron deficient compounds.* Ph.D. thesis, University of Indiana, Bloomington, IN. 1974. 520 p.