

Acta Cryst. (1962). **15**, 1058

Unit cell and space group of technetium carbonyl, $Tc_2(CO)_{10}$. By DANIEL WALLACH, *Department of Chemistry, University of California, Los Angeles 24, California, U.S.A.*

(Received 24 April 1962)

Colorless, diamond-shaped crystals of $Tc_2(CO)_{10}$ were prepared by Hileman, Huggins & Kaesz (1961). Tc^{99} , the isotope used, emits weak β radiation. $Tc_2(CO)_{10}$ was studied for comparison with $Mn_2(CO)_{10}$ and $Re_2(CO)_{10}$, the other carbonyls of the same period, which are of interest because of their metal-metal bonds (Dahl, Ishishi & Rundle, 1957).

A single crystal of $Tc_2(CO)_{10}$ was lodged in a capillary which served to absorb the β -radiation from Tc^{99} and to prevent decomposition. All photographs were taken with unfiltered Cu radiation. Measurements of b_0 and c_0 were made on zero-level Weissenberg photographs calibrated with CeO_2 powder patterns ($a = 5.411$, $\lambda(CuK\alpha) = 1.542$ Å). The value of a_0 was estimated from a rotation photograph and the angle β was calculated from measurements of a first-level Weissenberg photograph by the method described by Buerger (1942). The unit cell dimensions for $Tc_2(CO)_{10}$ are compared in the table with the values found by Dahl, Ishishi & Rundle for $Mn_2(CO)_{10}$ and $Re_2(CO)_{10}$.

| | $Mn_2(CO)_{10}$ | $Tc_2(CO)_{10}$ | $Re_2(CO)_{10}$ |
|-------------|--------------------------|--------------------------|--------------------------|
| a_0 | 14.16 Å | 14.73 ± 0.05 Å | 14.70 Å |
| b_0 | 7.11 | 7.22 ± 0.02 | 7.15 |
| c_0 | 14.67 | 14.90 ± 0.02 | 14.91 |
| β | 105° | 104.6 ± 0.1 | 106° |
| Space group | <i>Ia</i> or <i>I2/a</i> | <i>Ia</i> or <i>I2/a</i> | <i>Ia</i> or <i>I2/a</i> |

The two possible monoclinic space groups, *Ia* and *I2/a*, correspond to the observed conditions for reflections:

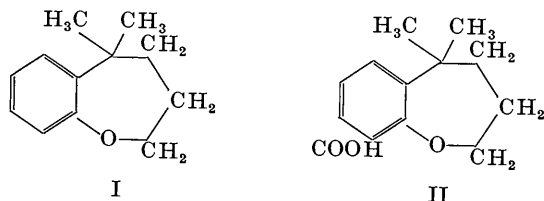
$$hkl: h+k+l=2n; \quad h0l: h=2n.$$

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The crystal data for 5:5-dimethyl homochroman and 5:5-dimethyl homochroman carboxylic acid. By M. SUNDARALINGAM and G. A. JEFFREY, *The Crystallography Laboratory, The University of Pittsburgh, Pittsburgh 13, Pa., U.S.A.*

(Received 14 May 1962)

Single crystals of two homochroman derivatives were obtained from specimens kindly provided by Dr H. Hart of Michigan State University. The compounds and their crystal data are as follows:



5:5-dimethyl homochroman, $C_{12}H_{16}O$, I, recrystallized from cyclopentane as monoclinic plates; space group

The measured density of 2.11 g.cm.^{-3} corresponds to four ($p_c = 2.14 \text{ g.cm.}^{-3}$) $Tc_2(CO)_{10}$ molecules in the unit cell. Visual comparison of intensities with those on photographs of crystals of $Mn_2(CO)_{10}$ and $Re_2(CO)_{10}$ indicated that $Tc_2(CO)_{10}$ is isomorphous with these other crystals, as the cell dimensions strongly suggest. Thus there seems no doubt that technetium pentacarbonyl is dimeric, as are the pentacarbonyls of Mn and Re.

The unconventional body-centered monoclinic cell was used for comparison with the other carbonyls, which were reported in this way. The cell constants for space group *Cc* or *C2/c* are as follows:

$$a'_0 = 17.56, \quad b'_0 = 7.20, \quad c'_0 = 14.73 \text{ Å}, \quad \text{and } \beta = 128^\circ$$

where

$$c' = a, \quad a' = a + c.$$

No further work on these crystals is contemplated in these laboratories.

I am grateful for the assistance of Linda Forrest, Mary Burke, Dr Håkon Hope and Dr K. N. Trueblood. This work was done with the support of an Undergraduate Fellowship from the National Science Foundation.

References

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 DAHL, L., ISHISHI, E. & RUNDLE, R. E. (1957). *J. Chem. Phys.* **26**, 1750.
 HILEMAN, J., HUGGINS, D. & KAESZ, H. D. (1961). *J. Amer. Chem. Soc.* **83**, 2953.

$P2_1/c$ from systematic extinctions, ($h0l$) absent l odd, ($0k0$) absent k odd; $a = 12.20$, $b = 6.97$, $c = 12.53 \pm 0.05$ Å, $\beta = 95.4^\circ$; $Z = 4$; $D_x = 1.10 \text{ g.cm.}^{-3}$; $D_m = 1.03 \text{ g.cm.}^{-3}$, by flotation in aqueous KBr.

5:5-dimethyl homochroman carboxylic acid, $C_{13}H_{16}O_3$, II, recrystallized from benzene as orthorhombic prisms; space group *Pbca*, from systematic extinctions, ($0kl$) absent k odd, ($h0l$) absent l odd, ($hk0$) absent k odd; $a = 12.42$, $b = 17.12$, $c = 11.52 \pm 0.05$ Å; $Z = 8$, $D_x = 1.19 \text{ g.cm.}^{-3}$; $D_m = 1.14 \text{ g.cm.}^{-3}$, by flotation in aqueous KBr.

No further work on these compounds is in progress. We are grateful to Dr Arnett of the Chemistry Dept., University of Pittsburgh for bringing these compounds to our attention and to the U.S. Public Health Service for the support of the research.