

Acta Cryst. (1965). **18**, 562**Crystal data for some dicyclopentadienyldinickel alkyne compounds.** By O. S. MILLS and B. W. SHAW, *Department of Chemistry, University of Manchester, Manchester 13, England*

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It has been shown (Tilney-Bassett & Mills, 1959; Tilney-Bassett, 1961) that dicyclopentadienyldinickel dicarbonyl reacts with acetylenes in a manner similar to dicobalt octacarbonyl (Sternberg, Greenfield, Friedel, Wotiz, Markby & Wender, 1954; Greenberg, Sternberg, Friedel, Wotiz, Markby & Wender, 1956). It was suggested that the molecular structures of these nickel compounds would be comparable to the corresponding cobalt derivatives, the structure of one of which has been determined (Sly, 1959).

In the course of our verification of these proposals we have determined the unit-cell dimensions of six derivatives and these are given in Table 1. Measurements were made on precession photographs taken with either molybdenum $K\alpha$ radiation ($\lambda=0.7107 \text{ \AA}$) or with cobalt $K\alpha$ radiation ($\lambda=1.7902 \text{ \AA}$). Densities were determined by suspension in aqueous solutions of potassium iodide.

The determination of the structure of derivative *A* has been completed. No further work on the remaining compounds is contemplated.

References

- GREENFIELD, H., STERNBERG, H. W., FRIEDEL, R. A., WOTIZ, J., MARKBY, R. & WENDER, I. (1956). *J. Amer. Chem. Soc.* **78**, 120.
 SLY, W. G. (1959). *J. Amer. Chem. Soc.* **81**, 18.
 STERNBERG, H. W., GREENFIELD, H., FRIEDEL, R. A., WOTIZ, J., MARKBY, R. & WENDER, I. (1954). *J. Amer. Chem. Soc.* **76**, 1457.
 TILNEY-BASSETT, J. F. (1961). *J. Chem. Soc.* p. 577.
 TILNEY-BASSETT, J. F. & MILLS, O. S. (1959). *J. Amer. Chem. Soc.* **81**, 4757.

Table 1. *Crystal data for dicyclopentadienyldinickel alkynes*

Derivative	<i>A</i>	<i>B</i>	<i>C</i>	<i>D</i>	<i>E</i>	<i>F</i>
Space group	$Pna2_1$	$P2_1/n$	$P2_1$ or $P2_1/m$	$P2_12_12_1$	$P1$ or $P\bar{1}$	$P1$ or $P\bar{1}$
Unit cell (\AA)						
<i>a</i>	17.73(5)	23.87(6)	6.15(3)	17.60(5)	10.90(4)	5.73()
<i>b</i>	9.49(3)	5.75(3)	12.37(4)	20.17(5)	11.57(4)	10.71()
<i>c</i>	11.62(4)	10.63(4)	8.75(3)	8.67(3)	18.99(4)	13.08()
α					118° 47'(5)	101° 45'
β		92° 36'(5)	100° 36'(5)		91° 4'(5)	108° 57'
γ					90° 30'(5)	90° 2'
D_m (g.cm ⁻³)	1.45(2)	1.50(2)	1.52(2)	1.50(2)	1.41(2)	1.57(2)
D_x (g.cm ⁻³)	1.45(1)	1.50(1)	1.53(1)	1.50(1)	1.42(1)	1.57(1)
<i>Z</i>	4	4	2	4	2	2
Radiation used	Cobalt	Molybdenum	Cobalt	Molybdenum	Molybdenum	Molybdenum
	<i>A.</i> $\text{C}_2\text{Ni}_2\text{C}_6\text{H}_5\text{C}_2\text{C}_6\text{H}_5$		<i>D.</i> $\text{C}_2\text{Ni}_2\text{C}_6\text{H}_5\text{C}_2\text{C}_6\text{H}_5\text{Ni}_2\text{C}_2$			
	<i>B.</i> $\text{C}_2\text{Ni}_2\text{HC}_2\text{N-C}_4\text{H}_9$		<i>E.</i> $\text{C}_2\text{Ni}_2\text{C}_6\text{H}_5\text{C}_2\text{C}_6\text{H}_5$			
	<i>C.</i> $\text{C}_2\text{Ni}_2\text{CH}_3\text{C}_2\text{CH}_3$		<i>F.</i> $\text{C}_2\text{Ni}_2\text{C}_6\text{H}_5\text{C}_2\text{H}$			

Acta Cryst. (1965). **18**, 562**Bond lengths and thermal vibrations in orthorhombic sulfur.** By AIMERY CARON and JERRY DONOHUE
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Abrahams (1961) has recently re-refined the structure of orthorhombic sulfur, using an improved form factor curve (Dawson, 1960*a, b*) and scale factors differing by 15 to 24% from the one used in his earlier work (Abrahams, 1955). In this note, Abrahams gave two widely differing sets of r.m.s. thermal displacements. One set, labelled 1960*A*, was obtained by assigning unit weights to observed reflections and weights of 0.1 to the unobserved reflections for which the F_o were set at $\frac{1}{2}F_{\text{min}}$. In obtaining the second set, labelled 1960*C*, the Hughes (1941)

weighting scheme was used, with $4F_{\text{min}} = 100$ and weights of 0.05 for the unobserved reflections. Individual positional and thermal parameters and their standard errors were not given; values of R were not mentioned. Abrahams suggested that there was correlation among the thermal parameters and thus implied that the thermal displacement values which he presented should not be discussed from a physical standpoint.

We feel, however, that the 1960*A* weighting scheme is indefensible, a conclusion supported by common sense,