Dimethoxyphenyl) hexanoyl chloride (1 g), prepared with oxalyl chloride in the same way as δ-(2,5-dimethoxybenzoyl)valeroyl chloride, was refluxed with p-toluidine (2 g) in dry benzene (50 ml) for 15 min. After cooling the solution was washed with water, dilute hydrochloric acid and bicarbonate solution. The solution was dried and evaporated and the residue was recrystallised from benzene--light petroleum and gave white needles m.p. 103.5—105°C. (Found: C 73.26; H 7.98; N N 3.79. $C_{21}H_{27}O_3N$ requires C 73.86; H 7.97; N 4.10.)

1,4-Bis (2-hydroxy-5-methoxybenzoyl) butane. 1,4-Bis(2,5-dimethoxybenzoyl)butane (3 g) was dissolved in a cooled solution of $AlCl_3$ (12 g) in nitrobenzene (150 ml). After 45 h at room temperature the mixture was decomposed with ice and the nitrobenzene was removed by steam distillation. The solid product was dissolved in chloroform and shaken with sodium hydroxide solution (1 N). The alkaline solution was acidified with hydrochloric acid and the crystals which separated were dissolved in chloroform. After drying the chloroform solution was run through a silica gel column and evaporated. A small amount of pure 1,4 - bis(2 - hydroxy - 5 - methoxybenzoyl)butane was obtained. It was recrystallised from ethanol-benzene, yellow prisms, m.p. 157—158°C. It gave a violet colour with $FeCl_3$ in methanol. (Found: C 66.49; H 6.22; OCH_3 15.9. $C_{20}H_{22}O_6$ requires C 67.03; H 6.19; OCH_3 16.1.)

An attempt to prepare this substance from adipoyl chloride, hydroquinone monomethylether and aluminium chloride using the same conditions as in the preparation of 1,4-bis (2,5-dimethoxybenzoyl)butane gave the bis-p-methoxyphenylester of adipic acid, white needles from ethanol, m.p. 126—127°C. (Found: C 67.13; H 6.04. $C_{20}H_{22}O_6$ requires C 67.02, H 6.19.)

Infrared spectrum of 1,4-bis(2-hydroxy-5-methoxybenzoyl)butane indicated that the demethylation took place in the *ortho* positions to the carbonylgroups. There was no absorption attributable to a hydroxyl group in the 3 500—2 500 cm⁻¹ region. The same is true for o-hydroxyacetophenone which is attributed to intramolecular hydrogen bonding by Hergert [6].

The carbonyl frequency of 1,4-bis(2,5-dimethoxybenzoyl) butane was measured to 1 665 cm⁻¹ and that of 1,4-bis(2-hydroxy-5-methoxybenzoyl) butane to 1 650 cm⁻¹. The difference is 15 cm⁻¹. The difference between the carbonyl frequencies of o-methoxyacetophenone and o-hydroxyacetophenone [6] is 14 cm⁻¹, from 1 649 cm⁻¹ to 1 635 cm⁻¹.

The low carbonyl frequency of 1,4-bis(2-hydroxy-5-methoxybenzoyl)butane may be explained if a conjugated chelate system as in o-hydroxyacetophenone [6] is assumed.

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Metal-Metal Bonding in a Mixed Chromium Molybdenum Oxide Phase of Rutile Structure

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Mixed chromium molybdenum oxide $(Cr,Mo)O_2$ of rutile type has been prepared by heating mixtures of chromium(III)-oxide, molybdenum(IV)oxide and molybdenum (VI)oxide $(Cr_2O_3 + aMoO_2 + MoO_3)$ in sealed, evacuated silica tubes and keeping the temperature at about 1 000°C for several days. For values of a of $3-7.5$ the X-ray powder patterns showed that the products only contained the rutile type phase. The width of the homogeneity range was not determined. No indications were observed of the metal atoms being ordered. The following unit cell dimensions were obtained:

	a	c	V
$Cr_{0.33}Mo_{0.67}O_2$	4.696 Å	2.886 Å	63.64 Å³
$Cr_{0.22}Mo_{0.78}O_2$	4.749	2.858	64.44
$Cr_{0.19}Mo_{0.81}O_2$	4.760	2.848	64.53

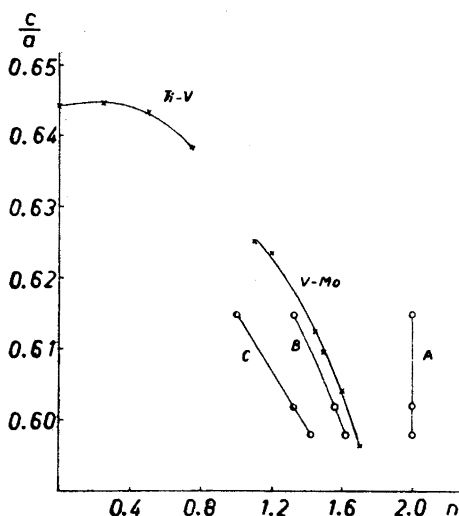The increase of the unit cell volume with the molybdenum content is in accordance

Fig. 1.

with the corresponding data for chromium dioxide ($V = 57.06$ Å^3 for rutile type cell [1]) and molybdenum dioxide ($V = 65.03$ Å^3 for subcell of distorted rutile type [2]). The decrease, with the molybdenum content, of the c axis (i.e. the shortest metal-metal distance of the structure) is the reverse of what would be expected from the relative sizes of the component atoms. This is taken as an indication of bonding forces acting between the neighbouring metal atoms of the mixed oxide as has previously been found for molybdenum dioxide (shortest metal-metal distance = 2.50 Å[2,3]) and several other compounds of transition metals when in valency states lower than their maximum ones [4]. The long metal-metal distance in chromium dioxide ($c = 2.917$ Å) [1] shows that no such bonding is present in this substance. This interpretation of the meaning of the intermetallic distances is supported by the magnetic data reported for the chromium and molybdenum dioxides [5]. It is also of interest to note that the metal atoms are "non-bonding" in the $Cr_2Cl_9^{3-}$ ion [6] while structural and magnetic data indicate bond formation in the $W_2Cl_9^{3-}$ ion [7,8].

It was shown in a previous article [4] that the axial ratio c/a provides an acceptable "normalized" measure of the metal-metal distance and this makes possible a comparison of rutile structures containing atoms of different sizes. On plotting the axial ratio against the number of excess valency electrons available per close metal-metal contact (n) (i.e. the number of valency electrons of the transition metal atoms which are not engaged in metal oxygen bonding) smooth curves are obtained.

Fig. 1 shows the curves thus derived for rutile type phases occurring in the systems $(Ti,V)O_2$ and $(V,Mo)O_2$. A few rutile type preparations in the systems $(V,Re)O_2$, $(V,Mo,Re)O_2$ and $(Nb,Mo)O_2$ give further data in fair agreement with the $(V,Mo)O_2$ curve or its extrapolation towards higher n values. For the $(Cr,Mo)O_2$ preparations, the following alternatives are indicated in the figure:

A. The excess valency electrons of both chromium and molybdenum are available for the formation of metal-metal bonds.

B. The excess chromium electrons are "non-bonding". All the metal atoms are tetravalent.

C. The excess chromium electrons are "non-bonding". The chromium atoms are trivalent and the corresponding proportion of the molybdenum atoms pentavalent or hexavalent.

It appears that curve B is in the best agreement with the data for other rutile phases containing molybdenum. This may be taken as an indication that the excess chromium electrons do not contribute to the metal-metal bonding and that the chromium atoms are probably in a tetravalent state.

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