

Table 1. Uronic acids eluted from anion exchange columns.

Tube	M HAc	Galacturonic acid	Guluronic acid	Mannuronic acid	Glucuronic acid
89-96	0.90-0.95	+			
97-103	0.95-1.00	+	+		
104-111	1.00-1.05		+		
139-150	1.20-1.25			+	
151-175	1.25-1.40				+

1. Derungs, R. and Deuel, H. *Helv. Chim. Acta* **37** (1954) 42.
2. Khym, J. X. and Doherty, D. G. *J. Am. Chem. Soc.* **74** (1952) 3199.
3. Hallén, A. *Acta Chem. Scand.* **14** (1960) 2249.
4. Davies, C. W. *Biochem. J.* **45** (1949) 38.
5. Haug, A. and Larsen, B. *Acta Chem. Scand.* **15** (1961) 1395.
6. Dubois, M., Gilles, K. A., Hamilton, J. K., Rebers, P. A. and Smith, F. *Anal. Chem.* **28** (1956) 350.

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### Crystal Data of $\text{MnO}_2 \cdot \text{P}_2\text{O}_5 \cdot \text{Cl}_4 \cdot (\text{CH}_3\text{COOC}_2\text{H}_5)_2$

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Bassett and Taylor<sup>1,2</sup> investigated 40 years ago the reactions between metal oxides and phosphorus oxychloride. Their aim was to establish the molecular structure of apatite and related compounds.

They discovered that the oxides of divalent manganese, zinc, magnesium and calcium react with phosphorus oxychloride to yield crystalline products. To a crystal chemist of to day this is somewhat surprising and these compounds deserve a structural investigation, as they certainly are not related to apatite.

Bassett and Taylor discovered also that  $\text{POCl}_3$  dissolved in acetone or ethylacetate or other related compounds reacts violently with the metal oxides mentioned above.

Hydrogen chloride is evolved during the reaction and the oxides are dissolved. After cooling a crystalline product appears, e.g.  $\text{MnO}_2 \cdot \text{P}_2\text{O}_5 \cdot \text{Cl}_4 \cdot (\text{CH}_3\text{COOC}_2\text{H}_5)_2$ .

All the compounds mentioned are rather hygroscopic, and we selected the manganese compound for an X-ray investigation as it was apparently less hygroscopic than the others. The chemical analysis confirmed the results of Bassett and Taylor: (Found: Cl 28.84; P 12.38. Calc. for  $\text{MnO}_2 \cdot \text{P}_2\text{O}_5 \cdot \text{Cl}_4 \cdot (\text{C}_4\text{H}_8\text{O}_2)_2$ : Cl 28.45; P 12.42).

A crystal of the dimensions: 1.7 mm  $\times$  0.8 mm  $\times$  0.4 mm was sealed in a Lindemann tube. Weissenberg and precession photographs have been taken using both Cu and Mo radiation.

The crystal proved to be monoclinic. The unit cell has the following dimensions:

$$\begin{aligned} a &= 14.27 \text{ \AA} \\ b &= 13.87 \text{ \AA} \\ c &= 10.04 \text{ \AA} \\ \beta &= 95^\circ 56' \end{aligned}$$

The following spectra were absent:  $h0l$  for  $h = 2n + 1$ ,  $0k0$  for  $k = 2n + 1$ . Thus the space group is  $P2_1/a$ . The density of the crystal is found as 1.671. The general point in  $P2_1/a$  is fourfold so the molecular weight of the asymmetric unit is 500. The calculated molecular weight for  $\text{MnO}_2 \cdot \text{P}_2\text{O}_5 \cdot \text{Cl}_4 \cdot (\text{C}_4\text{H}_8\text{O}_2)_2$  is 499.

A full structure determination of this compound has been started.

1. Bassett, H. and Taylor, H. S. *J. Chem. Soc.* **99** (1911) 1402.
2. Bassett, H. and Taylor, H. S. *Z. anorg. Chem.* **73** (1912) 75.

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