



The latter workers, basing their results on two-dimensional X-ray data alone, adduced evidence in favour of its composition being  $2\text{KC}_4\text{H}_3\text{O}_4 \cdot \text{C}_4\text{H}_4\text{O}_4$ . In view of the rarity of such a mixed crystal, it was considered worth while to determine the structure more accurately, in order both to establish conclusively its chemical composition and to fix more precisely the coordinates of the atoms, including the hydrogen atoms.

### Experimental

The new lattice parameters, based on a least-squares fit of neutron diffraction data ( $\lambda = 1.031 \text{ \AA}$ ), and intercomparison with X-ray data, are:

$$\begin{aligned} a &= 8.603 (8.631) \text{ \AA} & \alpha &= 78^\circ 20' (78^\circ 20') \\ b &= 7.475 (7.483) & \beta &= 112^\circ 20' (112^\circ 30') \\ c &= 6.902 (6.947) & \gamma &= 107^\circ 55' (107^\circ 55') \\ Z &= 2, \text{ space group } P\bar{1}. \end{aligned}$$

The figures in parentheses are the values of Gupta & Sahu (1970). There are two molecules of potassium hydrogen fumarate  $\text{KC}_4\text{H}_3\text{O}_4$  in general positions while the fumaric acid molecule itself is on a centre of symmetry at  $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$ . 701 independent X-ray reflexions were collected with  $\text{Cu K}\alpha$  radiation using Weissenberg photography, and their intensities were estimated visually.

### Refinement

Using the coordinates of Gupta & Sahu (1970), the data were refined by full-matrix least-squares analysis giving unit weight to each reflexion and using the modified version of the program *ORFLS* of Martin, Busing & Levy (1962). The refinement converged to an  $R$  value of 0.085. It was terminated when the shifts in the parameters were less than  $0.5 \sigma$ . A three-dimensional difference Fourier synthesis was used to locate the positions of the hydrogen atoms, and

Table 2. Atomic coordinates and their *e.s.d.*'s ( $\times 10^4$ ).

The results of Gupta & Sahu (1970) are shown in parentheses.

	$x/a$	$\sigma(x/a)$	$y/b$	$\sigma(y/b)$	$z/c$	$\sigma(z/c)$
C(1)	622 (622)	12 (19)	302 (305)	10 (20)	2685 (2768)	15 (23)
C(2)	294 (344)	12 (18)	2234 (2307)	10 (20)	2497 (2488)	20 (24)
C(3)	-1232 (-1268)	11 (18)	2512 (2486)	11 (21)	2011 (1731)	18 (25)
C(4)	-1521 (-1497)	12 (17)	4463 (4426)	11 (21)	1880 (1956)	20 (22)
C(5)	4754 (4768)	12 (17)	4287 (4286)	11 (18)	5656 (5685)	19 (21)
C(6)	5418 (5487)	12 (17)	2572 (2581)	11 (19)	6300 (6322)	18 (23)
O(1)	2160 (2148)	8 (12)	264 (297)	7 (14)	3272 (3247)	12 (16)
O(2)	-615 (-551)	8 (13)	-1070 (-1057)	7 (14)	2180 (2297)	13 (17)
O(3)	-2933 (-2936)	8 (12)	4742 (4689)	8 (14)	1102 (1111)	13 (16)
O(4)	87 (94)	8 (13)	5748 (5698)	8 (14)	2561 (2525)	14 (19)
O(5)	5229 (5274)	9 (12)	1478 (1481)	8 (14)	7785 (7820)	12 (15)
O(6)	6290 (6333)	9 (12)	2372 (2413)	8 (14)	5186 (5212)	13 (15)
K	4016 (4019)	2 (4)	2014 (2015)	2 (4)	715 (721)	4 (5)
H(1)	1480		3279		2835	
H(2)	-2319		1421		1082	
H(3)	-273		6988		2735	
H(4)	3809		4367		6353	
H(5)	7074		1409		5781	

Table 3. Anisotropic temperature factors and their *e.s.d.*'s ( $\times 10^5$ ).

The anisotropic temperature factor is in the form

$$T = \exp [-(B_{11}h^2 + \dots + 2B_{12}hk + \dots)].$$

Isotropic temperature factors are shown for the hydrogen atoms.

	$B_{11}$	$B_{22}$	$B_{33}$	$B_{12}$	$B_{13}$	$B_{23}$
C(1)	893 (152)	721 (129)	1560 (369)	260 (102)	383 (199)	334 (168)
C(2)	827 (154)	793 (137)	2040 (390)	335 (110)	641 (192)	-12 (176)
C(3)	760 (141)	631 (116)	1869 (365)	230 (097)	966 (171)	-484 (155)
C(4)	708 (152)	1155 (157)	2162 (408)	275 (114)	121 (207)	173 (200)
C(5)	746 (139)	1025 (143)	1840 (339)	487 (126)	425 (182)	12 (183)
C(6)	591 (144)	1027 (155)	1632 (338)	229 (115)	419 (191)	-423 (198)
O(1)	940 (108)	941 (099)	2139 (270)	519 (083)	678 (134)	11 (123)
O(2)	890 (114)	754 (098)	2255 (296)	298 (081)	146 (150)	-450 (128)
O(3)	750 (105)	1023 (104)	2577 (274)	399 (080)	734 (130)	-243 (131)
O(4)	725 (110)	953 (102)	2681 (323)	178 (077)	189 (157)	-489 (133)
O(5)	1285 (129)	1072 (103)	1849 (261)	509 (092)	1016 (153)	620 (139)
O(6)	994 (120)	1062 (110)	2155 (257)	556 (096)	784 (145)	394 (136)
K	618 (033)	821 (036)	1420 (089)	267 (022)	380 (036)	224 (036)
H(1)	3.8					
H(2)	6.7					
H(3)	5.6					
H(4)	3.7					
H(5)	3.8					

structure factor calculations, with hydrogen contributions included, reduced  $R$  to 0.078.

### Results

The list of structure factors is given in Table 1. The final coordinates of the atoms are given in Table 2 with the earlier results of Gupta & Sahu (1970) in parentheses. The thermal parameters of the atoms are given in Table 3. The bond lengths and angles are given in Table 4.

Table 4. Bond lengths and angles

Bond lengths		e.s.d.
C(1)–C(2)	1.524 (1.543) Å	0.011 (022)° Å
C(2)–C(3)	1.305 (1.320)	0.005 (023)
C(3)–C(4)	1.531 (1.562)	0.010 (022)
C(1)–O(1)	1.230 (1.231)	0.004 (019)
C(1)–O(2)	1.225 (1.246)	0.009 (019)
C(4)–O(3)	1.190 (1.215)	0.010 (012)
C(4)–O(4)	1.294 (1.276)	0.007 (012)
C(5)–C(6)	1.495 (1.495)	0.010 (020)
C(5)–C(5')	1.333 (1.354)	0.017 (051)
C(6)–O(5)	1.215 (1.218)	0.012 (013)
C(6)–O(6)	1.295 (1.300)	0.009 (014)
C(2)–H(1)	1.05	
C(3)–H(2)	1.03	
C(5)–H(4)	1.11	
O(4)–H(3)	1.00	
O(6)–H(5)	1.05	
Bond angles		e.s.d.
C(1)–C(2)–C(3)	123.84 (118.07)°	0.57 (1.33)°
C(2)–C(3)–C(4)	122.67 (115.97)	0.61 (1.15)
O(1)–C(1)–O(2)	124.43 (126.35)	0.80 (1.33)
O(1)–C(1)–C(2)	116.20 (113.33)	0.70 (1.00)
O(2)–C(1)–C(2)	119.20 (119.50)	0.67 (1.17)
O(4)–C(4)–O(3)	125.02 (125.53)	0.80 (1.17)
O(4)–C(4)–C(3)	113.16 (114.70)	0.62 (1.03)
O(3)–C(4)–C(3)	121.73 (116.70)	0.62 (1.03)
C(5)–C(6)–O(5)	119.63 (120.47)	0.63 (1.12)
C(5)–C(6)–O(6)	115.85 (114.52)	0.73 (1.08)
O(5)–C(6)–O(6)	122.85 (124.64)	0.63 (1.10)
C(6)–C(5')–C(5)	121.75 (123.50)	0.60 (1.20)
O(4)–H(3)–bO(2)	166.83	
O(6)–H(5)–bO(1)	167.95	

### Conclusion

The evidence produced earlier by Gupta & Sahu (1970) has been confirmed by this analysis. The molecular dimensions

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**A refinement of the crystal structure of durene.** By C. H. STAM, *Laboratory for Crystallography, Nieuwe Prinsengracht 126, Amsterdam, The Netherlands*

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The crystal structure of durene has been refined by means of three-dimensional Cu  $K\alpha$  counter data.

### Introduction

The crystal structure of durene (1,2,4,5-tetramethylbenzene) was determined by Robertson (1933) from projections. In the light of a crystal structure determination of 1,2,4,5-tetra-*t*-butylbenzene (van Bruynsvort, Eilermann, van der Meer & Stam, 1968) it was of interest for reasons

of the two kinds of 'molecules' ( $\text{KC}_4\text{H}_3\text{O}_4$  and  $\text{C}_4\text{H}_4\text{O}_4$ ) are significantly different ( $> 2\sigma$ ). The dimensions of the molecule in the special position  $\frac{1}{2}, \frac{1}{2}, \frac{1}{2}$  show this to be a molecule of fumaric acid because of the un-ionized carboxyl groups in it, the bond lengths and angles agreeing fairly well with values reported for the fumaric acid molecule by Brown (1966) and Bednowitz & Post (1966), Table 5. Moreover, the positions of the hydrogen atoms and their distances from the nearest oxygen atoms show quite conclusively which of them are of the type O(H) and which carboxyl groups are ionized or unionized. The correct chemical composition in this crystal is therefore  $2\text{KC}_4\text{H}_3\text{O}_4 \cdot \text{C}_4\text{H}_4\text{O}_4$ .

Table 5. Comparison of molecular dimensions of the acid molecule in the special position

	Brown (1966)	Bednowitz & Post (1966)	This work
C–C	1.46 Å	1.49 Å	1.49 Å
C=C	1.36	1.32	1.33
C–O···H	1.29	1.29	1.29
C=O	1.23	1.23	1.22
$\angle$ O–C–C	116.7°	116.0°	115.9°
$\angle$ O=C–C	119.0	119.5	119.6
$\angle$ O=C–O	124.3	124.4	122.9
$\angle$ C–C–C	122.8	122.5	121.8

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