Instrumental Achievements

Crystal Structure of 2,2-Dimethyl Succinic Acid

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The title compound crystallizes triclinically in space group of $P\bar{1}$. The C_2-COOH and C_3-COOH molecular groups are planar. The crystal structure is stabilized by the formation of intermolecular (O-H···O) hydrogen bonds.

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Succinic acid and its derivates have been used in the leather industry to improve the water repellency and wet strength;[1,2] to improve the froth in the flotation of various ores;[3] as vehicles for printing inks;[4] as flavoring in the food industry[5] and a gelating agent for marmalada;[6] as a coalescing agent for emulsion paints;[7] and for a wide variety of other industrial applications.[8–11]

The powder form of the title compound was purchased from Sigma Chemical Company. Prism-shaped single crystal were grown by slow evaporation in water at room temperature. In order to elucidate the structure Fig. 1, an X-ray crystal structure analysis was undertaken, and the results are presented here (Tables 1 – 3).

Figure 2 shows the molecular structure of the title compound ($C_6H_{10}O_4$). The C_3-COOH and C_2-COOH molecular groups are almost perfectly planar. The dihedral angle between the two carboxyl groups is 87.2(2)˚. The structural parameters of those two molecular groups are consistent with those in related compounds, *rac*-2,3-dibromosuccinic acid and sarcosinium trifluoroacetate.[12,13] The geometric parameters are listed in Table 2.

Some slight differences in the parameters of the title compound and the other related compounds may be explained by different intra and intermolecular contacts.[12,13]

Two inter-molecular hydrogen bonds were obtained in the crystal structure [$O_1\cdots O_4^{i}$, 2.642(2); $O_4\cdots O_3^{ii}$, 2.683(2)Å].

References

1. U. S. Pat. 2,795,517 (June 11, **1957**), A. Lowe and G. Wilkams (to Imperial Chemical Industries, Ltd.).

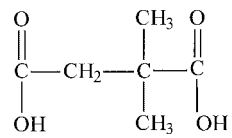

Fig. 1 Chemical structure.

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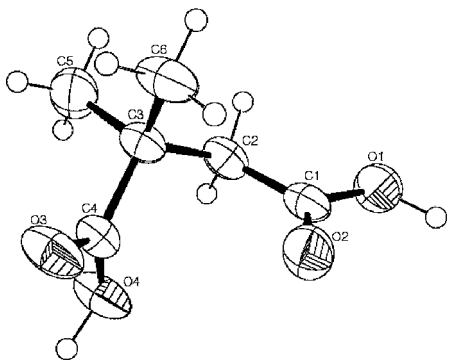

Fig. 2 Molecular structure of the title complex.

Table 1 Crystal and experimental data

Formula: [$C_6H_{10}O_4$]
Formula weight = 146.14
Space group: $P\bar{1}$ (No: 2)
Crystal system: triclinic, Z = 2
Lattice constants (Å) a = 5.661(2) b = 6.385(1) c = 11.461(2)
α = 74.51(1)˚ β = 78.69(2)˚ γ = 67.86(1)˚
V = 367.6(2)Å^3
D_x = 1.320 g/cm^3
μ(Mo K$_\alpha$) = 0.112 mm^{-1}
T = 293 K
Color: colorless
Radiation Mo K$_\alpha$ (λ = 0.71073 Å)
θ_{max} = 26.26˚
No. of reflection = 1472
No. of reflection used = 1199 ($I > 2\sigma(I)$)
R = 0.0366
Rw = 0.1030
$(\Delta\rho)_{max}$ = 0.190 eÅ^{-3}
$(\Delta\rho)_{min}$ = –0.202 eÅ^{-3}
Measurement: Enraf-Nonius CAD-4
Program system: SHELX97
Structure determinetion: SHELXS97
Refinement: full matrix least-square SHELXL97
Treatment of hydrogen atoms: geometric calculation

Table 2 Final atomic coordinates, equivalent isotropic thermal parameters (Å^2)

Atom	*x*/*a*	*y*/*b*	*z*/*c*	B_{eq}
O1	0.1676(3)	0.1605(2)	0.0220(1)	4.76(4)
O2	0.4910(2)	0.0818(2)	0.1288(1)	4.65(4)
O3	0.5214(2)	0.2396(2)	0.3993(1)	4.71(4)
O4	0.2392(2)	0.0678(2)	0.4130(1)	4.86(4)
C1	0.2636(3)	0.1800(2)	0.1115(1)	3.47(4)
C2	0.0724(3)	0.3291(3)	0.1912(1)	3.55(5)
C3	0.1817(3)	0.4248(2)	0.2690(1)	3.42(4)
C4	0.3324(3)	0.2315(2)	0.3654(1)	3.25(4)
C5	-0.0469(3)	0.5848(3)	0.3380(2)	5.11(6)
C6	0.3477(3)	0.5624(3)	0.1901(2)	4.66(6)
H1	0.302(5)	0.062(5)	-0.033(3)	
H2A	-0.02771(0)	0.24037(0)	0.24512(0)	
H2B	-0.04414(0)	0.45858(0)	0.14031(0)	
H4	0.3260(5)	-0.036(5)	0.480(3)	
H5A	-0.14818(0)	0.70969(0)	0.28049(0)	
H5B	0.01581(0)	0.64617(0)	0.38850(0)	
H5C	-0.15056(0)	0.49816(0)	0.38785(0)	
H6A	0.24935(0)	0.68299(0)	0.12995(0)	
H6B	0.49461(0)	0.46102(0)	0.15033(0)	
H6C	0.40331(0)	0.62962(0)	0.24043(0)	

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j)$.

Table 3 Some geometrical parameters with estimated standard deviations (Å, °)

O1-C1	1.304(2)	O4-C4	1.291(2)
O2-C1	1.231(2)	C1-C2	1.487(2)
O3-C4	1.231(2)	C4-C3	1.523(2)
O1-C1-C2	113.8(1)	C1-C2-C3	116.0(1)
O2-C1-O1	122.9(1)	C2-C3-C5	107.5(1)
O3-C4-O4	123.0(1)	C4-C3-C2	111.0(1)
O3-C4-C3	121.6(1)	C6-C3-C2	111.3(1)
O1-C1-C2-C3	–160.2(1)	O4-C4-C3-C6	–161.1(1)
O2-C1-C2-C3	20.6(2)	O4-C4-C3-C5	79.9(2)
O3-C4-C3-C6	22.7(2)	C1-C2-C3-C4	–67.2(2)
O3-C4-C3-C2	146.9(1)	C1-C2-C3-C6	56.7(2)
O3-C4-C3-C5	–96.3(2)	C1-C2-C3-C5	176.6(1)
O4-C4-C3-C2	–36.9(2)		

2. L. Seligberger, *J. Am. Leather Chemist' Assoc.*, **1967**, *62*, 346.
3. U. S. Pat. 2,689,043 (Sept. 14, **1954**), W. M. Fisher (to Minerec. Corp.).
4. U. S. Pat. 2,778,806, (Jan. 22, **1957**), W. M. Hatckinson (to Phillips Petroleum Co.).
5. T. Take and H. Itsuka, *Kaseigaku Zasshi*, **1966**, *17*, 213.
6. Neth Pat. Appl. 6, 607, 121, (Feb. 5, **1967**) (to Pferffer&Lougen K6).
7. Brit Pat. 1,033,466 (June 22, **1966**), J. J. Haitson (to Distilks Co., Ltd.).
8. U. S. Pat. 2,362,041 (Nov. 7, **1944**), J. S. Rerchert *et al*. (to E. I. du Pont de Nemours Co.).
9. U. S. Pat. 2,374,187 (Apr. 24, **1945**), L. H. Flett (to Allied Chemical And Dye Corp.).
10. U. S. Pat. 2,793,348 (May 14, **1957**), R. D. Ayles Woth (to Emery Industries, Inc.).
11. U. S. Pat. 2,743,293 (Nov. 26, **1956**), M. De Groote (to Petrolite Corp.)
12. M. Bolte and A. Degen, *Acta Cryst*., **2000**, *C56*, Cix and e410.
13. V. H. Rodrigues, J. A. Paixao, M. M. R. R. Costa, and A. Motos Beja, *Acta Cryst*., **2000**, *C56*, 1053.