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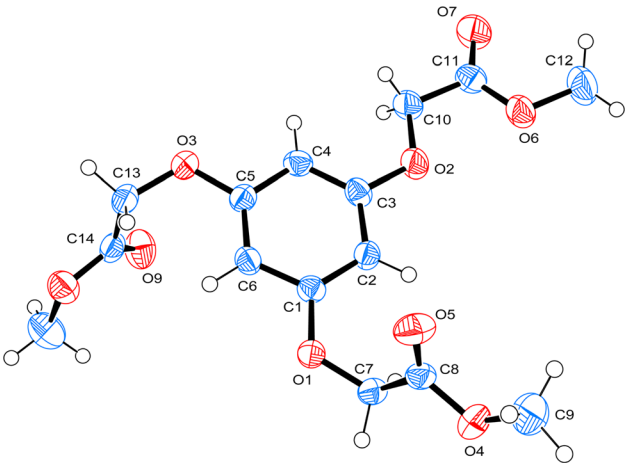
# The crystal structure of trimethyl 2,2′,2″-(benzene-1,3,5-triyltris(oxy))triacetate, $C_{15}H_{18}O_9$

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**Abstract**

$C_{15}H_{18}O_9$, triclinic, $P\bar{1}$ (no. 2), $a$ = 8.4962(7) Å, $b$ = 8.6835(7) Å, $c$ = 11.9767(10) Å, $\alpha$ = 90.9650(10)°, $\beta$ = 94.9460(10)°, $\gamma$ = 115.9310(10)°, $V$ = 790.21(11) Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.0318, $wR_{ref}(F^2)$ = 0.0855, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colorless block |
| Size: | 0.25 × 0.23 × 0.21 mm |
| Wavelength: | Mo *Kα* radiation (0.71073 Å) |
| $\mu$: | 0.12 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX-II, $\varphi$ and $\omega$ |
| $\theta_{max}$, completeness: | 26.0°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 8728, 3099, 0.015 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\sigma(I_{obs})$, 2828 |
| $N(param)_{refined}$: | 221 |
| Programs: | Bruker [1], SHELX [2, 3], WinGX/ORTEP [4] |

## Source of material

All chemicals were purchased from commercial sources and used without further purification. The 2,2′,2″-(benzene-1,3,5-triyltris(oxy))triacetic acid (5 mmol, 1.501 g) was dissolved in anhydrous methanol (50 mL), then add 5 drops of anhydrous tin tetrachloride. The mixture was refluxed for 8 h. When the reaction vessel had cooled to room temperature, the reaction mixture was filtered. The solvent was removed by evaporation *in vacuo*, the title product was obtained by recrystallization from methanol.

## Experimental details

All H-atoms bonded to C atoms were placed geometrically and refined using a riding model with common isotropic displacement factors $U_{iso}$(H) = 1.2 or 1.5 $U_{eq}$(parent C-atom).

## Comment

The phenoxyacetic acid and its esters are plant growth regulators and herbicides [5, 6]. They are mainly used as compounds, which can be modified to find new active compounds. Unlike benzoic acid and fatty acids, phenoxyacetic acid derivatives have flexible and varied coordination modes and spatial conformations, and other structural characteristics [7, 8]. Thus, the synthesis and crystal structure study of the title compound are of great significance.

Single-crystal structure analysis revealed that the title compound crystallized in the triclinic space group $P\bar{1}$. The ORTEP diagram is presented in Figure. Bond lengths and angles are all in the expected ranges. The bond length of O1–C7, O2–C10, O3–C13 are 1.4149(14), 1.4115(15),

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**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.52503 (11) | 0.70930 (10) | 0.50102 (7) | 0.0397 (2) |
| O2 | 0.63951 (13) | 1.14824 (11) | 0.24293 (8) | 0.0482 (3) |
| O3 | 1.02660 (11) | 1.24728 (11) | 0.57614 (7) | 0.0421 (2) |
| O4 | 0.22899 (12) | 0.39083 (12) | 0.29436 (8) | 0.0477 (2) |
| O5 | 0.52030 (12) | 0.54099 (12) | 0.29972 (9) | 0.0508 (3) |
| O6 | 0.54858 (14) | 1.21785 (12) | 0.04808 (8) | 0.0508 (3) |
| O7 | 0.74230 (16) | 1.49418 (13) | 0.06792 (9) | 0.0649 (3) |
| O8 | 0.96858 (13) | 1.12425 (12) | 0.85781 (8) | 0.0462 (2) |
| O9 | 0.86020 (14) | 1.27747 (14) | 0.75727 (8) | 0.0555 (3) |
| C1 | 0.62078 (15) | 0.87017 (14) | 0.46557 (9) | 0.0315 (2) |
| C2 | 0.57818 (15) | 0.93173 (14) | 0.36800 (9) | 0.0339 (3) |
| H2 | 0.477872 | 0.864181 | 0.320249 | 0.041* |
| C3 | 0.68998 (16) | 1.09846 (15) | 0.34291 (9) | 0.0342 (3) |
| C4 | 0.83886 (16) | 1.20146 (14) | 0.41271 (10) | 0.0352 (3) |
| H4 | 0.912146 | 1.312084 | 0.394558 | 0.042* |
| C5 | 0.87659 (15) | 1.13509 (15) | 0.51142 (9) | 0.0328 (3) |
| C6 | 0.77001 (15) | 0.97087 (14) | 0.53843 (9) | 0.0336 (3) |
| H6 | 0.796996 | 0.927772 | 0.604089 | 0.040* |
| C7 | 0.36280 (15) | 0.59877 (15) | 0.43992 (10) | 0.0353 (3) |
| H7A | 0.299643 | 0.664766 | 0.416927 | 0.042* |
| H7B | 0.292133 | 0.512744 | 0.488577 | 0.042* |
| C8 | 0.38512 (15) | 0.51064 (14) | 0.33782 (10) | 0.0341 (3) |
| C9 | 0.2291 (3) | 0.2954 (2) | 0.19511 (14) | 0.0704 (5) |
| H9A | 0.262084 | 0.370214 | 0.134353 | 0.106* |
| H9B | 0.113549 | 0.203994 | 0.175545 | 0.106* |
| H9C | 0.311793 | 0.248377 | 0.209148 | 0.106* |
| C10 | 0.73863 (18) | 1.32012 (15) | 0.21726 (11) | 0.0418 (3) |
| H10A | 0.861718 | 1.344456 | 0.219399 | 0.050* |
| H10B | 0.728460 | 1.396129 | 0.273453 | 0.050* |
| C11 | 0.67627 (17) | 1.35388 (16) | 0.10326 (11) | 0.0410 (3) |
| C12 | 0.4900 (2) | 1.2438 (2) | −0.06459 (13) | 0.0650 (4) |
| H12A | 0.431381 | 1.316162 | −0.060333 | 0.097* |
| H12B | 0.410085 | 1.135098 | −0.101503 | 0.097* |
| H12C | 0.589792 | 1.297453 | −0.106345 | 0.097* |
| C13 | 1.06662 (15) | 1.19376 (16) | 0.68125 (10) | 0.0385 (3) |
| H13A | 1.189042 | 1.265329 | 0.707941 | 0.046* |
| H13B | 1.050442 | 1.076366 | 0.672214 | 0.046* |
| C14 | 0.95169 (15) | 1.20471 (15) | 0.76702 (10) | 0.0373 (3) |
| C15 | 0.8712 (2) | 1.1341 (2) | 0.94821 (13) | 0.0658 (4) |
| H15A | 0.910775 | 1.251990 | 0.973129 | 0.099* |
| H15B | 0.889932 | 1.071978 | 1.009410 | 0.099* |
| H15C | 0.748106 | 1.084864 | 0.922318 | 0.099* |

1.4145(14) Å, respectively, which are similar to those reported in the literature [9, 10].

Molecules of the title compound form a set of weak C–H···O intermolecular hydrogen bonds in the crystal phase (Table 3). The geometric characteristics of these hydrogen bonds are close that does not allow to separate out a main structural motif of the crystal packing.

**Table 3:** Symmetry operations and geometric characteristics of intermolecular hydrogen bonds.

| Hydrogen bond | Symmetry operation | Geometrical characteristics | | |
|---|---|---|---|---|
| | | H···A, Å | D···A, Å | D–A···, Å |
| C2–H···O9 | 1 − *x*, 2 − *y*, 1 − *z* | 2.65 | 3.529 (1) | 157 |
| C7–H7a···O9 | 1 − *x*, 2 − *y*, 1 − *z* | 2.57 | 3.379 (2) | 141 |
| C9–H9b···O8 | 1 − *x*, 1 − *y*, 1 − *z* | 2.64 | 3.300 (2) | 127 |
| C10–H10b···O5 | *x*, 1 + *y*, *z* | 2.62 | 3.385 (2) | 137 |
| C12–H12a···O7 | 1 − *x*, 3 − *y*, −*z* | 2.65 | 3.604 (3) | 175 |
| C13–H13a···O5 | 2 − *x*, 2 − *y*, 1 − *z* | 2.31 | 3.233 (1) | 158 |

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**Conflict of interest statement:** The authors declare no conflicts of interest regarding this article.

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