

Fig. 1. Projections of the structure of copper decanoate along the a and b axes. Large circles are copper atoms, small open circles are oxygen atoms and small closed circles are carbon atoms.

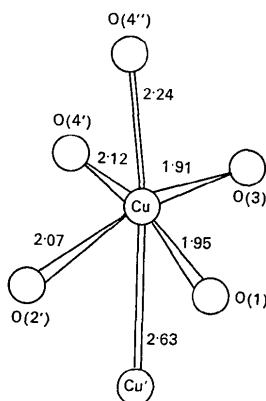


Fig. 2. Coordination of copper atoms in copper decanoate. The bond lengths are given in Å and have standard deviations of approximately 0.05 Å.

One of the pairs of hydrocarbon chains in each dimer has the carbon and oxygen atoms arranged in the common plane zigzag conformation throughout its length but the chains in the other pair are distorted near the carboxyl group by rotations about the bonds C(11)–C(12) and C(12)–C(13). The remainder of the atoms in these 'bent' chains, however, are arranged in the usual plane zigzag conformation, and pack together with those of the straight chains to form a structure which may be described by a triclinic subcell with dimensions $a=4.42$, $b=5.28$, $c=2.56$ Å; $\alpha=70.2$, $\beta=109.6$, $\gamma=117.5^\circ$. These subcell dimensions are close to those reported in several other hydrocarbon chain compounds (see *e.g.* Lomer, 1963) and it is interesting that this close packing of the chains over part of their length is realized despite the distortion of some of them which is apparently necessary for the copper and oxygen atoms to achieve their favoured arrangement.

None of the C–C bond lengths or C–C–C bond angles differed significantly from the commonly accepted values, the mean observed values being 1.57 ± 0.02 and $110.2 \pm 0.5^\circ$. The temperature factors of the carbon atoms in the methyl groups are very large. Similarly high temperature factors for atoms near the methyl group ends of hydrocarbon chains were observed in copper butyrate and also by Lewis & Lomer (1969) in potassium decanoate. It is not clear whether these high values are due to large thermal motions or to a degree of static disorder among the chains.

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Anhydrous Copper(II) Octanoate

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(Received 27 August 1974; accepted 20 September 1974)

Abstract. $\text{Cu}[\text{CH}_3(\text{CH}_2)_6\text{COO}]_2$, triclinic, $P\bar{1}$, $a=8.80$ (2), $b=5.20$ (1), $c=22.06$ (1) Å; $\alpha=95.1$ (5), $\beta=92.4$ (5), $\gamma=110.5$ (5)°, $Z=2$, $M=350$; $D_c=1.230$ (6),

$D_m=1.21$ (4) g cm⁻³. The copper atom environment is the same as that in copper decanoate but the packing of the hydrocarbon chains differs in detail.

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Introduction. Copper(II) octanoate was prepared by adding copper carbonate to an excess of solution of oc-

tanoic acid in ethanol, and stirring. When effervescence had ceased the precipitated copper octanoate was filtered off, washed with ethanol and dried. The precipitate was then dissolved in octanoic acid at 50°C and the solution allowed to cool and evaporate in an open dish placed in a vacuum chamber. Bundles of dark blue-green needle-shaped crystals appeared after a few weeks. The separation of a single crystal suitable for X-ray analysis was difficult because the crystals were fragile and easily deformed, and many were twinned. The experimental methods of the structure determination were the same as those described in the accompanying paper by Lomer & Perera (1974). 420 independent reflexions were measured out of a possible total of 1050. The final R value was 0.159. The atomic coordinates and temperature factors are given in Table 1.*

Table 1. *Atom parameters: coordinates as fractions of the unit-cell edges ($\times 10^3$) and temperature factors in \AA^2*

The standard deviations given in parentheses are in units corresponding to the least significant digit in the parameters.

| | x/a | y/b | z/c | B |
|-------|----------|----------|---------|-----------|
| C(1) | 198 (5) | 63 (9) | 81 (2) | 1.4 (10) |
| C(2) | 311 (7) | 78 (11) | 129 (2) | 6.0 (17) |
| C(3) | 291 (6) | 307 (9) | 163 (2) | 3.5 (13) |
| C(4) | 402 (6) | 297 (9) | 215 (2) | 4.1 (14) |
| C(5) | 398 (7) | 542 (10) | 250 (2) | 5.0 (15) |
| C(6) | 517 (6) | 532 (9) | 298 (2) | 3.9 (14) |
| C(7) | 503 (5) | 761 (8) | 335 (2) | 2.4 (12) |
| C(8) | 621 (6) | 752 (10) | 385 (2) | 4.3 (14) |
| C(9) | 626 (7) | 999 (9) | 419 (2) | 5.9 (16) |
| C(10) | 726 (9) | 990 (9) | 467 (2) | 10.4 (22) |
| C(11) | 788 (6) | 735 (9) | 44 (2) | 1.8 (17) |
| C(12) | 657 (6) | 557 (9) | 73 (2) | 2.4 (12) |
| C(13) | 745 (6) | 527 (9) | 124 (2) | 4.1 (15) |
| C(14) | 780 (6) | 807 (9) | 154 (2) | 3.6 (14) |
| C(15) | 886 (7) | 781 (9) | 202 (2) | 4.6 (15) |
| C(16) | 898 (7) | 30 (10) | 241 (2) | 5.4 (16) |
| C(17) | 3 (6) | 5 (9) | 291 (2) | 3.8 (14) |
| C(18) | 998 (7) | 247 (10) | 328 (2) | 6.2 (17) |
| C(19) | 114 (8) | 207 (12) | 380 (2) | 7.2 (18) |
| C(20) | 111 (10) | 453 (12) | 418 (3) | 11.1 (25) |
| O(1) | 93 (4) | 202 (6) | 69 (1) | 3.6 (9) |
| O(2) | 215 (4) | 876 (6) | 55 (1) | 3.3 (9) |
| O(3) | 752 (6) | 96 (6) | 36 (2) | 3.3 (9) |
| O(4) | 875 (6) | 604 (8) | 22 (1) | 2.3 (9) |
| Cu | 930 (1) | 200 (1) | 13 (1) | 2.9 (2) |

Discussion. Diagrams of the structure projected on to planes perpendicular to the a and b axes are shown in Fig. 1. The structure contains centrosymmetric dimers with the coordination of the copper and oxygen atoms, shown in Fig. 2, almost identical to that in copper butyrate (Bird & Lomer, 1972) and copper decanoate (Lomer & Perera, 1965). As in copper decanoate, one pair of hydrocarbon chains in each dimer

has the common plane zigzag conformation and the other pair of chains is distorted by rotations about the bonds C(9)–C(10) and C(10)–C(11). However, as shown in Fig. 1 the undistorted parts of the 'bent' chains do *not* lie with their axes parallel to those of the straight chains. The structure of the octanoate is thus much more like that of the butyrate than that of the decanoate. This similarity is also revealed by comparison of the cell dimensions. If the unit-cell axes given by Bird & Lomer (1972) for the butyrate are re-labelled to conform with the convention usual in long-chain compounds, that the longest axis is the c axis, the dimensions of the butyrate cell become $a=9.004$, $b=5.16$, $c=11.736$ Å, $\alpha=85.3$, $\beta=95.2$, $\gamma=108.7^\circ$. The short dimensions and the included angle γ are close

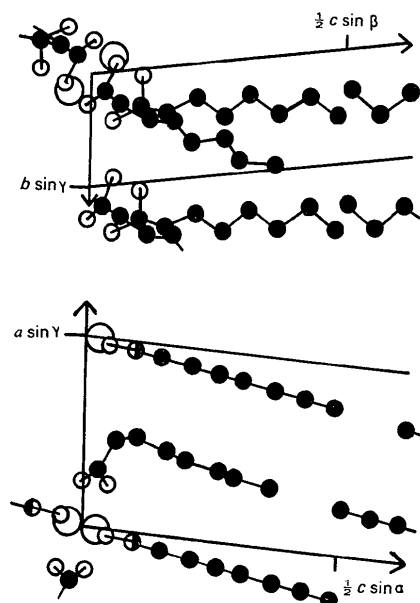


Fig. 1. Projections of the structure of copper octanoate along the a and b axes. Large circles are copper atoms, small open circles are oxygen atoms and small closed circles are carbon atoms.

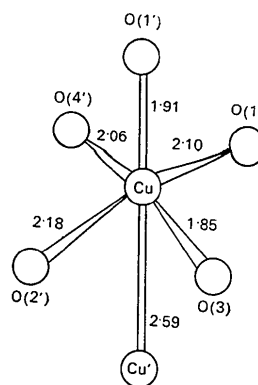


Fig. 2. Coordination of copper atoms in copper octanoate. The bond lengths are given in Å, and have standard deviations of approximately 0.05 Å.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30684 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

to those of the octanoate but differ significantly from those of the decanoate. It is not clear whether the difference between the structure of the decanoate and the other two copper alkanoates arises from the different method of preparation of the decanoate crystals or whether it is a consequence of the longer chain length.

As in copper decanoate none of the bond lengths or angles in the hydrocarbon chain differ significantly from the commonly accepted values, the observed mean values being 1.53 ± 0.04 Å and $113 \pm 3^\circ$. The large temperature factors associated with several of the carbon atoms correspond to r.m.s. displacements of the order of 0.8 Å, which is rather high for random ther-

mal motions of the atoms. It is thus possible that the displacements are due to static disorder in this crystal, but the exact nature of the displacements and the precise crystal structure cannot be determined until better methods of growing good-quality single crystals become available.

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3-Methyl-2,4-dicarbomethoxy- Δ 3-cephem

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Abstract. Monoclinic, $P2_1/c$, $a = 6.708$ (9), $b = 17.75$ (2), $c = 10.59$ (1) Å, $\beta = 97.0$ (1)°, $C_{11}H_{13}NO_5S$, $Z = 4$, $M = 271.3$, $D_x = 1.439$ g cm⁻³, racemic, m.p. 113-115°C. The configuration is (2*RS*, 5*RS*, 6*SR*). The nitrogen atom of the β -lactam ring is 0.196 Å above the plane of its ligands. The methoxycarbonyl group in position 4 makes an angle of 40.4° with the Δ 3-double-bonding system and the resulting steric hindrance with the methyl group in position 3 causes the sp^2 -planes of C(2) and C(3) to be inclined at 7.9° to each other.

Einführung. Die Kristalle wurden von Herrn Dr Kühlein (Hoechst AG) hergestellt.

Die Röntgenintensitäten erfassten wir auf einem Siemens-Einkristalldiffraktometer [on-line Betrieb; Fünfwertmessung (Hoppe, 1965); Mo $K\alpha$ -Strahlung; von 2970 theoretisch zu vermessenden Reflexen erreichten 2337 in einer Grobmessung eine vorgegebene Mindestintensität und wurden zu Ende vermessen; 1851 Reflexe hatten einen aus der Zählstatistik ermittelten relativen Fehler $r < 15\%$; systematische Auslöschungen: $0k0: k = 2n + 1$, $h0l: l = 2n + 1$]. Das Strukturproblem konnte mit Hilfe der Methoden zur direkten Phasenbestimmung (Germain & Woolfson, 1968; Germain, Main & Woolfson, 1970) gelöst werden. Die Strukturparameter wurden nach der Methode der Kleinsten-Quadrate (volle Matrix) verfeinert. Die Wasserstoffatome liessen sich aus einer Differenz-Fouriersynthese der Elektronendichte in chemisch sinnvoller Lage ermitteln. Der endgültige R -Faktor ($R = \frac{\sum |F_o| - |F_c|}{\sum |F_o|} \times 100\%$)* ist 4,1% (2337 Reflexe). Die zehn

Tabelle 1. Atomkoordinaten und isotrope Temperaturkoeffizienten für $C_{11}H_{13}NO_5S$

Die isotropen Temperaturkoeffizienten stellen die Werte vor der anisotropen Verfeinerung der Temperaturkoeffizienten dar. Den Wasserstoffatomen sind die um 1 Å² erhöhten Temperaturkoeffizienten der Atome zugeordnet, an denen sie gebunden sind. Die Standardabweichungen sind in Klammern in Einheiten der letzten Dezimalstelle aufgeführt.

| | x | y | z | B [Å ²] |
|-------|-------------|-------------|-------------|---------------------|
| S(1) | 0,33847 (8) | 0,13040 (3) | 0,31879 (6) | 3,15 (4) |
| O(1) | 0,2103 (3) | 0,3590 (1) | 0,4366 (2) | 5,1 (1) |
| O(2) | 0,8351 (2) | 0,1097 (1) | 0,4251 (1) | 3,9 (1) |
| O(3) | 0,7305 (2) | 0,0264 (1) | 0,2737 (1) | 3,9 (1) |
| O(4) | 0,6702 (3) | 0,3926 (1) | 0,2281 (2) | 4,7 (1) |
| O(5) | 0,6689 (2) | 0,3931 (1) | 0,4372 (1) | 3,4 (1) |
| N(1) | 0,4503 (2) | 0,2639 (1) | 0,4201 (2) | 2,6 (1) |
| C(1) | 0,5793 (3) | 0,1458 (1) | 0,2588 (2) | 2,6 (1) |
| C(2) | 0,6526 (3) | 0,2262 (1) | 0,2606 (2) | 2,7 (1) |
| C(3) | 0,5826 (3) | 0,2803 (1) | 0,3305 (2) | 2,6 (1) |
| C(4) | 0,3961 (3) | 0,1883 (1) | 0,4584 (2) | 2,8 (1) |
| C(5) | 0,2126 (4) | 0,2236 (1) | 0,5100 (2) | 3,4 (2) |
| C(6) | 0,2767 (3) | 0,2969 (1) | 0,4524 (2) | 3,1 (1) |
| C(7) | 0,7318 (3) | 0,0934 (1) | 0,3297 (2) | 2,8 (1) |
| C(8) | 0,8576 (5) | -0,0309 (2) | 0,3408 (3) | 5,0 (2) |
| C(9) | 0,8205 (3) | 0,2382 (1) | 0,1801 (2) | 3,3 (2) |
| C(10) | 0,6445 (3) | 0,3609 (1) | 0,3239 (2) | 2,9 (1) |
| C(11) | 0,7354 (5) | 0,4707 (1) | 0,4389 (3) | 4,4 (2) |
| H(1) | 0,562 (4) | 0,129 (1) | 0,164 (3) | 3,6 |
| H(2) | 0,499 (4) | 0,167 (2) | 0,514 (2) | 3,8 |
| H(3) | 0,225 (4) | 0,224 (2) | 0,601 (3) | 4,4 |
| H(4) | 0,082 (4) | 0,208 (2) | 0,472 (3) | 4,4 |
| H(5) | 0,856 (5) | -0,072 (2) | 0,288 (3) | 6,0 |
| H(6) | 1,003 (5) | -0,014 (2) | 0,353 (3) | 6,0 |
| H(7) | 0,802 (5) | -0,036 (2) | 0,430 (3) | 6,0 |
| H(8) | 0,772 (4) | 0,231 (2) | 0,090 (3) | 4,3 |
| H(9) | 0,920 (4) | 0,202 (2) | 0,201 (3) | 4,3 |
| H(10) | 0,880 (4) | 0,283 (2) | 0,186 (3) | 4,3 |
| H(11) | 0,874 (5) | 0,472 (2) | 0,417 (3) | 5,4 |
| H(12) | 0,732 (4) | 0,486 (2) | 0,536 (3) | 5,4 |
| H(13) | 0,648 (5) | 0,498 (2) | 0,400 (3) | 5,4 |

* Die Atomformfaktoren und die entsprechenden realen und imaginären Korrekturen für die anormale Dispersion wurden der Literatur (*International Tables for X-ray Crystallography*, 1962) entnommen.