

increase in metal size is certainly expected with an increase in coordination number. How the cation can influence the stoichiometry and coordination number is difficult to assess because complete results were not reported for $[(\text{CH}_3)_4\text{N}]_4[\text{Er}(\text{NCS})_7]$. Packing interactions in $[(\text{C}_4\text{H}_9)_4\text{N}]_3[\text{Er}(\text{NCS})_6]$ appear normal and the closest distance between Er atoms is 12.37 Å. From the packing diagram in Fig. 2 it appears that $[\text{Er}(\text{NCS})_6]^{3-}$ ions are surrounded by aliphatic carbon chains from $(\text{C}_4\text{H}_9)_4\text{N}^+$ cations. The smaller $(\text{CH}_3)_4\text{N}^+$ cations would be expected to leave the metal coordination sphere more exposed.

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Structure of Thallium Hydrogen Phthalate*

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Abstract. $[\text{Tl}(\text{C}_8\text{H}_5\text{O}_4)]$, $M_r = 369.43$, orthorhombic $P2_1ab$, $a = 6.615$ (2), $b = 10.047$ (2), $c = 12.878$ (2) Å, $V = 855.9$ Å³, $Z = 4$, $D_x = 2.868$ g cm⁻³, $\lambda(\text{Mo } K\alpha_1) = 0.70926$ Å, $\mu = 190.4$ cm⁻¹, $F(000) = 649.7$, room temperature, final $R = 0.039$ for 613 observed reflections [$I > 2\sigma(I)$] out of 821 independent reflections. Bond lengths and bond angles are within the normal range. The aromatic ring is planar within 0.01 Å. The planes of the carboxyl groups containing C(1) and C(8) are rotated out of plane from the aromatic ring by 67.9 and 29.3°, respectively.

Introduction. Thallium hydrogen phthalate (I), has been suggested as a possible monochromator for use in soft X-ray spectroscopy (Bohm & Ulmer, 1970). Compound (I) yields 3.7 times the intensity of potas-

sium hydrogen phthalate at the same wavelength and resolution in the region from about 10 to 100 Å (Okaya, 1965). Despite the interest generated by this important application, a single-crystal X-ray structure has not been previously reported.

Experimental. Crystals of (I) were obtained from Quartz Products Co., Plainfield, NJ, and were used as received. Crystal dimensions were 0.16 × 0.13 × 0.11 mm. CAD-4 diffractometer, θ -2 θ scans. θ -scan range $(0.8 + 0.34\tan\theta)^\circ$. Scan speed 1.0 to 8.2° min⁻¹. Background first and last one-sixth of scan range. Graphite-monochromated Mo $K\alpha$ radiation. Unit cell from 25 reflections, $14 \leq \theta \leq 21^\circ$. Empirical ψ -scan and spherical absorption corrections, transmission = 0.041–0.012. $(\text{Sin}\theta)/\lambda_{\text{max}} = 0.353$ Å⁻¹. Index range $-7 \leq h \leq 7$, $0 \leq k \leq 11$, $0 \leq l \leq 15$. Standard reflections 400 and 060 showed max. r.m.s. variation of 1.9% with no obvious trends. R_{int}

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Table 1. Fractional coordinates and equivalent isotropic thermal parameters (\AA^2) for thallium hydrogen phthalate

$$U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	U_{eq}
Tl(1)	0.25000	-0.10277 (8)	-0.04760 (6)	5.9
O(1)	0.190 (3)	0.137 (2)	0.097 (2)	6.7
C(1)	0.254 (10)	0.059 (2)	0.163 (2)	5.2
O(2)	0.394 (3)	-0.020 (2)	0.159 (1)	6.6
C(2)	0.124 (4)	0.052 (2)	0.266 (2)	4.7
C(3)	0.144 (4)	0.163 (2)	0.325 (2)	4.8
C(4)	0.042 (4)	0.167 (3)	0.427 (2)	6.0
C(5)	-0.072 (5)	0.056 (3)	0.456 (2)	6.7
C(6)	-0.086 (4)	-0.058 (3)	0.392 (2)	5.1
C(7)	0.014 (4)	-0.057 (2)	0.296 (2)	4.5
C(8)	-0.020 (4)	-0.176 (3)	0.229 (2)	4.8
O(3)	-0.008 (3)	-0.164 (2)	0.132 (1)	5.4
O(4)	-0.043 (3)	-0.288 (2)	0.275 (2)	5.8

Table 2. Selected distances (\AA) and angles ($^\circ$) for thallium hydrogen phthalate

Tl(1)—O(1)	3.07 (2)	Tl(1)—O(1 ^a)	2.72 (2)
Tl(1)—O(1 ^b)	3.00 (2)	Tl(1)—O(2)	2.95 (2)
Tl(1)—O(2 ^c)	3.02 (2)	Tl(1)—O(3)	2.94 (2)
O(1)—C(1)	1.23 (4)	O(2)—C(1)	1.22 (6)
O(3)—C(8)	1.25 (3)	O(4)—C(8)	1.29 (3)
C(1)—C(2)	1.58 (4)	C(2)—C(3)	1.35 (3)
C(2)—C(7)	1.37 (3)	C(3)—C(4)	1.48 (4)
C(4)—C(5)	1.39 (5)	C(5)—C(6)	1.41 (4)
C(6)—C(7)	1.40 (3)	C(7)—C(8)	1.50 (3)
C(6)—C(7)	1.40 (3)		

O(1)—Ti(1)—O(1 ^a)	151.2 (7)	O(1)—Ti(1)—O(1 ^b)	110.1 (6)
O(1)—Ti(1)—O(2)	43.3 (5)	O(1)—Ti(1)—O(2 ^c)	82.4 (5)
O(1)—Ti(1)—O(3)	67.0 (5)	O(1 ^a)—Ti(1)—O(1 ^b)	89.1 (5)
O(1 ^a)—Ti(1)—O(2)	122.0 (6)	O(1 ^a)—Ti(1)—O(2 ^c)	99.6 (5)
O(1 ^a)—Ti(1)—O(3)	84.2 (6)	O(1 ^b)—Ti(1)—O(2)	84.9 (5)
O(1 ^b)—Ti(1)—O(2 ^c)	134.6 (5)	O(1 ^b)—Ti(1)—O(3)	134.9 (5)
O(2)—Ti(1)—O(2 ^c)	124.4 (7)	O(2)—Ti(1)—O(3)	62.2 (5)
O(2 ^c)—Ti(1)—O(3)	90.4 (5)	O(1)—C(1)—O(2)	130 (3)
O(1)—C(1)—C(2)	115 (4)	O(2)—C(1)—C(2)	115 (2)
C(1)—C(2)—C(3)	112 (3)	C(1)—C(2)—C(7)	124 (2)
C(2)—C(3)—C(4)	118 (3)	C(3)—C(4)—C(5)	117 (3)
C(4)—C(5)—C(6)	122 (3)	C(5)—C(6)—C(7)	118 (2)
C(2)—C(7)—C(6)	121 (2)	C(2)—C(7)—C(8)	124 (2)
C(6)—C(7)—C(8)	116 (2)	O(3)—C(8)—O(4)	124 (2)
O(3)—C(8)—C(7)	119 (2)	O(4)—C(8)—C(7)	117 (2)

Symmetry code: (a) $x, -\frac{1}{2} + y, -z$; (b) $\frac{1}{2} + x, -y, -z$; (c) $-\frac{1}{2} + x, -y, -z$.

= 0.041 from merging equivalent reflections in original data set containing 1566 measured reflections, resulting in 613 observed reflections [$I > 2\sigma(I)$] out of 821 unique reflections. Structure solved by assuming the structure was isomorphous with the Rb salt (Smith, 1975). Least-squares refinement minimized $\sum w(\Delta F)^2$, $w = [\sigma_{\text{c}}^2(F) + 0.030F^2]^{-1}$, $\sigma_{\text{c}}^2(F)$ based on counting statistics. Scale factor, isotropic type-II extinction parameter (Zachariasen, 1967; Larson, 1967), positional parameters and anisotropic thermal parameters for heavy atoms were refined, H atoms not located, $R = 0.039$, $wR = 0.045$, $S = 2.027$, final $(\Delta/\sigma)_{\text{max}} = 0.027$. Final ΔF Fourier synthesis $-0.9 \leq \rho \leq 1.18 \text{ e \AA}^{-3}$. Six highest peaks in final Fourier all less than 1.5 \AA from Tl. Scattering factors, f , f' and

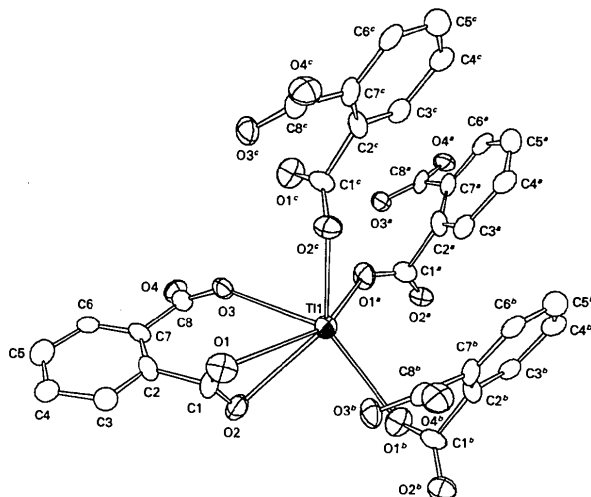


Fig. 1. ORTEP (Johnson, 1965) drawing of thallium hydrogen phthalate showing the atom-numbering scheme. Thermal ellipsoids are 35% probability.

f'' from *International Tables for X-ray Crystallography* (1974). Calculations on a Cray-1 using the Los Alamos Crystal Structure System developed primarily by A. C. Larson (Larson, 1977).*

Discussion. Final parameters are listed in Table 1. The numbering scheme is shown in Fig. 1. Each Tl^+ is surrounded by O atoms, with the six nearest at 2.72 (2), 2.94 (2), 2.95 (2), 3.00 (2), 3.02 (2) and 3.07 (2) \AA . The average value of these distances is comparable with that of the isomorphous structure of the Rb^+ salt (Smith, 1975) and 0.14 \AA greater than that of the K^+ salt (Okaya, 1965). The planes of the carboxyl groups containing C(1) and C(8) are rotated out of plane from the aromatic ring by 67.9 and 29.3 $^\circ$, respectively. Bond lengths and angles are given in Table 2.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52245 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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