used: Enraf–Nonius *SDP* (Frenz, 1980), *ORTEP*II (Johnson, 1976). The structure of the title compound is shown in Fig. 1. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1, selected bond distances and angles are listed in Table 2.*

Related literature. Ascochlorin (2) is a terpenoid antibiotic (Tamura, Suzuki, Takatsuki, Ando & Arima, 1968), and the structure was elucidated by X-ray analysis (Nawata, Ando, Tamura, Arima & Iitaka, 1969; Nawata & Iitaka, 1971). The title compound is a 4-*O*-carboxymethyl derivative of ascochlorin (Hosokawa, Matsuura, Takahashi, Ando & Tamura, 1990) and is an antidiabetic agent (Hosokawa, Ando & Tamura, 1985).

* Lists of structure factors, anisotropic thermal parameters, bond lengths, bond angles, torsion angles, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53532 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1991). C**47**, 686–687

Triethylammonium 3,3,6,6-tetrathioxocyclodi(phosphadithianate) at 178 K

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(*Received* 31 *July* 1990; *accepted* 20 *August* 1990)

Abstract. $[(C_2H_5)_3NH]_2[P_2S_8]$, $M_r = 522{\cdot}9$, monoclinic, $P2_1/c$, $a = 6{\cdot}929\ (3)$, $b = 13{\cdot}157\ (7)$, $c = 13{\cdot}447\ (5)$ Å, $\beta = 98{\cdot}07\ (3)°$, $V = 1213{\cdot}8$ Å^3, $Z = 2$, $D_x = 1{\cdot}43$ Mg m^{-3}, $F(000) = 520$, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}75$ mm^{-1}, $T = 178$ K, $R = 0{\cdot}026$ for 2396 reflections. The anion P_2S_4 ring is exactly centrosymmetric and possesses a chair conformation. The axial P—S bond is appreciably shorter than the equatorial (1·952, 1·987 Å). The anion and cation are connected by a hydrogen bond from the equatorial exocyclic S atom, S3···N 3·30 Å.

Experimental. A colourless prism 0·8 × 0·4 × 0·3 mm, obtained from acetonitrile solution, was mounted in inert oil and transferred to the cold gas stream of the diffractometer (Siemens *R*3 with LT-2 low temperature attachment). 4256 intensities were registered to $2\theta_{max} = 55°$ using monochromated Mo $K\alpha$ radiation (ω scans, width 1·1°, constant speed 7·3° min^{-1}, quadrant $-h+k+l$ and some $-k$ equivalents, index ranges h −8→0, k −16→16, l −17→17). Merging equivalents gave 2779 unique reflections (R_{int} 0·026), of which 2396 with $F > 4\sigma(F)$ were used for all calculations (program system Siemens *SHELXTL-Plus*). Three check reflections showed no significant intensity change. No absorp-

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^4)

	x	y	z	U_{eq}*
N	8372 (2)	1662 (1)	2141 (1)	206 (4)
C(1)	9529 (2)	1810 (1)	1287 (1)	253 (5)
C(2)	11094 (3)	1016 (2)	1251 (1)	346 (6)
C(3)	9511 (3)	1911 (1)	3153 (1)	258 (5)
C(4)	9843 (3)	3034 (1)	3310 (1)	325 (6)
C(5)	7442 (3)	630 (1)	2169 (1)	265 (5)
C(6)	6311 (3)	319 (1)	1170 (1)	335 (6)
P(1)	4783 (1)	3651 (1)	594 (1)	177 (1)
S(1)	2356 (1)	4653 (1)	298 (1)	202 (1)
S(2)	7176 (1)	4655 (1)	1022 (1)	209 (1)
S(3)	4445 (1)	3104 (1)	1933 (1)	244 (1)
S(4)	5089 (1)	2799 (1)	−559 (1)	263 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

0108-2701/91/030686-02$03.00

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Table 2. *Bond lengths* (Å) *and angles* (°)

P(1)—S(1)	2·130 (1)	P(1)—S(2)	2·135 (1)
P(1)—S(3)	1·984 (1)	P(1)—S(4)	1·948 (1)
S(1)—S(2^{i})	2·060 (1)	N—C(1)	1·504 (2)
N—C(3)	1·510 (2)	N—C(5)	1·505 (2)
C(1)—C(2)	1·511 (3)	C(3)—C(4)	1·505 (3)
C(5)—C(6)	1·513 (2)		

S(1)—P(1)—S(2)	103·3 (1)	S(1)—P(1)—S(3)	101·7 (1)
S(2)—P(1)—S(3)	99·6 (1)	S(1)—P(1)—S(4)	112·3 (1)
S(2)—P(1)—S(4)	113·8 (1)	S(3)—P(1)—S(4)	123·6 (1)
P(1)—S(1)—S(2^{i})	102·6 (1)	P(1)—S(2)—S(1^{i})	104·5 (1)
C(1)—N—C(3)	113·1 (1)	C(1)—N—C(5)	114·4 (1)
C(3)—N—C(5)	110·2 (1)	N—C(1)—C(2)	113·3 (1)
N—C(3)—C(4)	112·9 (1)	N—C(5)—C(6)	113·1 (1)

Symmetry operation: (i) $1-x, 1-y, -z$.

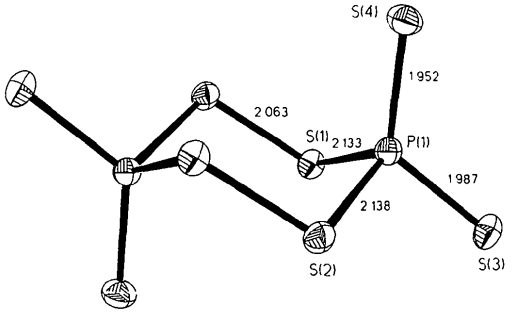

Fig. 1. Thermal ellipsoid plot (50% level) of the anion of the title compound, showing the numbering scheme of the independent atoms and the libration-corrected bond lengths.

tion correction. Cell constants were refined from setting angles of 50 reflections in the range 2θ 20–23°.

The structure was solved by routine direct methods and refined anisotropically on F to R 0·026, wR 0·034. H atoms were included using a riding model. The weighting scheme was $w^{-1} = \sigma^2(F) + 0{\cdot}00015F^2$. 109 parameters; S 1·6; max. Δ/σ 0·001; max., min. $\Delta\rho + 0{\cdot}39$, $-0{\cdot}25$ e Å^{-3}. Final atomic coordinates are presented in Table 1,* with derived bond lengths (uncorrected) and angles in Table 2. A rigid-body libration correction (Schomaker & Trueblood, 1968) was applied (R_{lib} 0·067); corrected bond lengths are shown in Fig. 1. A packing diagram is shown in Fig. 2.

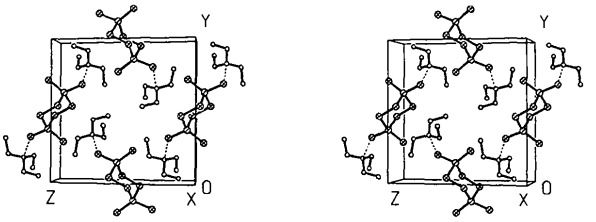

Fig. 2. Stereographic packing diagram of the title compound (H atoms omitted), showing the H bonds as dashed lines.

Related literature. The pyridinium salt of the same anion was studied by Minshall & Sheldrick (1978) at room temperature. The corrected bond lengths of the anions are almost identical in both studies.

We thank the Fonds der Chemischen Industrie for financial assistance and Professor H.-H. Falius for providing the sample.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53499 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1991). C**47**, 687–689

Structure of Cholest-5-en-4-one

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(*Received* 21 *March* 1990; *accepted* 8 *August* 1990)

Abstract. $C_{27}H_{44}O$, $M_r = 384{\cdot}7$, orthorhombic, $P2_12_12_1$, $a = 10{\cdot}326$ (1), $b = 10{\cdot}460$ (1), $c = 22{\cdot}331$ (1) Å, $V = 2412{\cdot}0$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}06$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 4{\cdot}3$ cm^{-1}, $F(000) = 856$, $R = 0{\cdot}061$, $wR = 0{\cdot}060$ for 2588 unique observed reflections with $F_o > 4\sigma(F_o)$. The A ring has

0108-2701/91/030687-03$03.00

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