

Absorption correction: semi-empirical
 $T_{\min} = 0.126$, $T_{\max} = 0.147$
 2468 measured reflections
 1835 independent reflections
 1809 observed reflections
 $[F \geq 4.0\sigma(F)]$

$k = -15 \rightarrow 0$
 $l = -13 \rightarrow 13$
 3 standard reflections
 frequency: 120 min
 intensity variation: 1%

Refinement

Refinement on F^2
 Final $R = 0.0161$
 $wR = 0.0229$
 $S = 1.170$
 1809 reflections
 137 parameters
 H atoms in fixed calculated positions
 $w = 1/[\sigma^2(F) + 0.0003F^2]$

$(\Delta/\sigma)_{\max} = 0.16$
 $\Delta\rho_{\max} = 0.75 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.44 \text{ e } \text{\AA}^{-3}$
 Extinction correction: isotropic
 Extinction coefficient: $\chi = 0.00111(7)$
 Atomic scattering factors from *SHELXTL/PC*

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Pd	-0.05429 (3)	-0.06461 (1)	0*	0.0238 (1)
Cl(1)	-0.19669 (10)	-0.20113 (5)	0.04311 (9)	0.0455 (2)
Cl(2)	-0.29969 (9)	-0.00235 (5)	-0.08551 (7)	0.0364 (2)
S(1)	0.18716 (9)	-0.11671 (5)	0.08628 (6)	0.0296 (2)
C(2)	0.30259 (34)	-0.01051 (22)	0.11925 (22)	0.0347 (3)
C(3)	0.30130 (34)	0.05968 (20)	0.03418 (27)	0.0332 (3)
S(4)	0.07538 (10)	0.07422 (5)	-0.01601 (8)	0.0268 (2)
C(5)	0.11814 (35)	0.10379 (21)	-0.15151 (22)	0.0332 (3)
C(6)	0.22475 (36)	0.03587 (21)	-0.21746 (22)	0.0322 (3)
S(7)	0.10553 (14)	-0.07130 (5)	-0.23429 (7)	0.0337 (2)
C(8)	0.26360 (36)	-0.13620 (21)	-0.31258 (23)	0.0374 (3)
C(9)	0.42448 (34)	-0.17397 (22)	-0.25808 (27)	0.0376 (3)
S(10)	0.39670 (12)	-0.27670 (5)	-0.18021 (7)	0.0359 (2)
C(11)	0.25769 (34)	-0.24026 (18)	-0.07290 (25)	0.0310 (3)
C(12)	0.33818 (33)	-0.16084 (19)	-0.01272 (24)	0.0326 (3)

*Coordinate fixed to define origin.

Table 2. Bond lengths (\AA) and angles ($^\circ$)

Pd—Cl(1)	2.325 (1)	Pd—Cl(2)	2.333 (1)
Pd—S(1)	2.261 (1)	Pd—S(4)	2.253 (1)
S(1)—C(2)	1.823 (3)	S(1)—C(12)	1.824 (3)
C(2)—C(3)	1.497 (4)	C(3)—S(4)	1.832 (3)
S(4)—C(5)	1.823 (3)	C(5)—C(6)	1.530 (4)
C(6)—S(7)	1.813 (3)	S(7)—C(8)	1.823 (3)
C(8)—C(9)	1.503 (4)	C(9)—S(10)	1.811 (3)
S(10)—C(11)	1.811 (3)	C(11)—C(12)	1.517 (4)
Cl(1)—Pd—Cl(2)	94.5 (1)	Cl(1)—Pd—S(1)	88.1 (1)
Cl(2)—Pd—S(1)	176.7 (1)	Cl(1)—Pd—S(4)	171.1 (1)
Cl(2)—Pd—S(4)	87.2 (1)	S(1)—Pd—S(4)	89.8 (1)
Pd—S(1)—C(2)	102.3 (1)	Pd—S(1)—C(12)	106.1 (1)
C(2)—S(1)—C(12)	99.4 (1)	S(1)—C(2)—C(3)	113.9 (2)
C(2)—C(3)—S(4)	110.0 (2)	Pd—S(4)—C(3)	105.5 (1)
Pd—S(4)—C(5)	112.0 (1)	C(3)—S(4)—C(5)	101.5 (1)
S(4)—C(5)—C(6)	118.0 (2)	C(5)—C(6)—S(7)	111.2 (2)
C(6)—S(7)—C(8)	100.9 (1)	S(7)—C(8)—C(9)	117.3 (2)
C(8)—C(9)—S(10)	117.8 (2)	C(9)—S(10)—C(11)	104.3 (1)
S(10)—C(11)—C(12)	112.4 (2)	S(1)—C(12)—C(11)	112.0 (2)

The title compound was prepared by refluxing PdCl_2 and $[\text{12}]_{\text{ane}}\text{S}_4$ in MeCN under N_2 for 4 h; the crystals were grown in MeCN. The data were collected with the crystal held in the cold stream of an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986) using ω - 2θ scans and the learnt-profile

method (Clegg, 1981). The structure was solved by interpretation of a Patterson synthesis which yielded the positions of the metal and one S atom. Refinement was by full-matrix least squares with all non-H atoms allowed anisotropic thermal motion. A determination of the polarity of the structure clearly favoured that adopted [$\eta = 1.02(4)$]. *SHELXTL/PC* (Sheldrick, 1990) was used to solve and refine the structure and to generate the figures and tables.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55465 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1010]

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Acta Cryst. (1993). **C49**, 168–171

Structure of the Organic Radical Cation Salt 2,3,6,7-Tetrakis(methylthio)naphthalene Hexafluoroarsenate

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(Received 5 May 1992; accepted 20 August 1992)

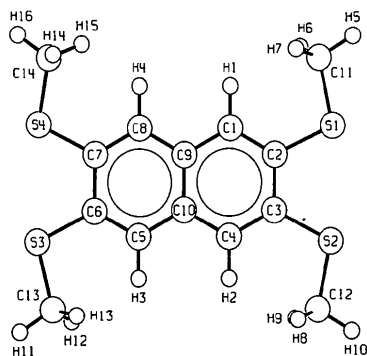
Abstract

Dimers of planar TMTN molecules [TMTN = 2,3,6,7-tetrakis(methylthio)naphthalene] form stacks along the *c* direction, surrounded by disordered AsF_6^- ions. The interplanar distances between the TMTN molecules are 3.275 (4) \AA (within the dimer) and 3.645 (4) \AA (between the dimers). The TMTN molecules within the dimer are related by a centre of symmetry.

Comment

This work is part of a series of studies into the structures and physical properties of electro-

chemically prepared cation radical salts (c.r.s.) with chalcogen-substituted aromatic hydrocarbons as donors. Four other salts based on substitutions on the naphthalene skeleton have already been studied in this series: tris[2,6-dimethoxy-3,7-bis(methylthio)naphthalene] diperchlorate, (2,6-DMbTN)₃(ClO₄)₂ (Noreland, Olovsson & Olovsson, 1992), tris[2,7-dimethoxy-3,6-bis(methylthio)naphthalene] diperchlorate, (2,7-DMbTN)₃(ClO₄)₂ (Olovsson & Olovsson, 1992a), tris(2,3,6,7-tetramethoxynaphthalene) bis(hexafluoroarsenate), (TMN)₃(AsF₆)₂ (Olovsson & Olovsson, 1991) and the corresponding perchlorate compound, (TMN)₃(ClO₄)₂ (Olovsson & Olovsson, 1992b).



TMTN

Bond angles in the AsF₆⁻ ion are 80.4–100.5° with e.s.d.'s 0.7–1.0°. The asymmetric unit consists of one TMTN molecule and one AsF₆⁻ ion. All atoms are in general positions. The TMTN molecule is slightly puckered with a dihedral angle of 2.5° between the least-squares planes defined by the two C rings in the molecule. Deviations from planarity in the TMTN molecule were also calculated relative to the least-squares plane defined by all ten ring C atoms C1–C10 [coplanar within 0.051 (4) Å]: C11 –0.076 (5), C12 –0.063 (6), C13 –0.428 (6), C14 –0.214 (5), S1 –0.133 (1), S2 0.014 (1), S3 –0.234 (1) and S4 0.128 (1) Å. The methyl groups are in this way displaced away from the centre of the dimer to avoid collisions with the neighbouring methyl groups (*cf.* Fig. 1). As expected, the atomic displacement parameters of the atoms in the methylthio groups are significantly larger than those of the other C atoms (*cf.* Fig. 1 and Table 1). The extremely large displacement parameters of the F atoms motivated the attempt to use a disordered model for the AsF₆⁻ ion. Since the displacement ellipsoid of the As atom is not unusually large, it was assumed to be ordered while introducing two positions each for all the F atoms.

Stacking arrangement. The TMTN molecules are packed in stacks along the *c* axis, with the AsF₆⁻ ions surrounding them (Fig. 1). Within a stack, the

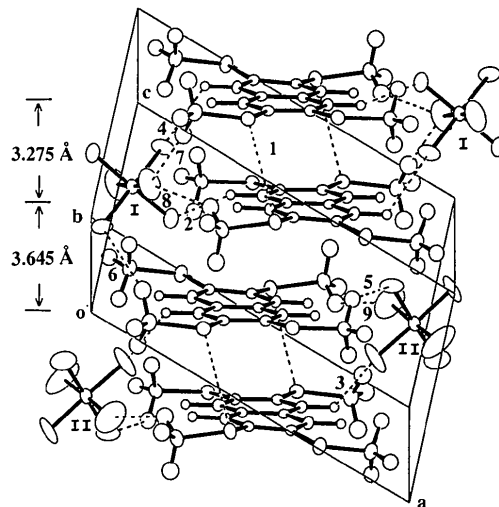


Fig. 1. A view of the molecular packing. The shortest S...S contact and the intermolecular distances ≤ 2.5 Å are indicated as dotted lines (*cf.* Table 3). The interplanar distances are between the least-squares planes of the TMTN molecules (defined by the ten ring C atoms). For each of the disordered AsF₆⁻ ions one of the possible orientations is shown [(I) F1A–F6A; (II) F1B–F6B]. All thermal ellipsoids are plotted at 20% probability.

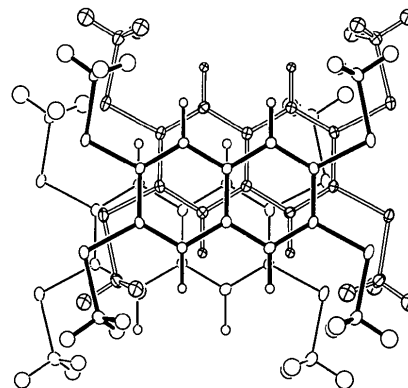


Fig. 2. Overlap between the TMTN molecules in the stack. The dimer consists of the molecules with thick filled bonds and unfilled bonds (related by a centre of symmetry). The molecule with thin filled bonds is the upper molecule in the next dimer along the stack.

TMTN molecules form dimers with interplanar distances 3.275 (4) Å (within the dimer) and 3.645 (4) Å (between the dimers). The molecular overlap is shown in Fig. 2. The molecules are *displaced* relative to each other within the dimer, in contrast to what has been found in the other substituted naphthalene salts, where they are *turned* relative to each other (Noreland *et al.*, 1992; Olovsson & Olovsson, 1991, 1992a,b). This displacement makes S...S contacts between molecules in adjacent stacks possible (*cf.* Table 3). It also prevents the formation of S...S

chains within the stack which were found in the 2,6-DMbTN and 2,7-DMbTN salts. There is only one S...S contact shorter than the van der Waals contact distance, 3.6 Å (Bondi, 1964): S2...S4 3.338 (2) Å (occurs twice within the dimer; cf. Fig. 1).

Experimental

Crystal data

C₁₄H₁₆S₄·AsF₆⁻

M_r = 501.43

Triclinic

*P*1

a = 12.458 (2) Å

b = 11.468 (1) Å

c = 7.105 (1) Å

α = 94.61 (1)°

β = 100.04 (1)°

γ = 112.24 (1)°

V = 913.3 (3) Å³

Z = 2

D_x = 1.823 Mg m⁻³

Mo *K*α radiation

λ = 0.71069 Å

Cell parameters from 25

reflections

θ = 16.5–19.5°

μ = 2.35 mm⁻¹

T = 296 K

Needle

0.22 × 0.11 × 0.08 mm

Black; greenish black metallic lustre

Data collection

Rigaku AFC-6R four-circle diffractometer

ω/2θ scans

Absorption correction: empirical

T_{min} = 0.836, *T_{max}* = 0.952

11063 measured reflections

10610 independent reflections

5487 observed reflections

[*F*² > σ(*F*²)]

θ_{max} = 30°

h = 0 → 17

k = -16 → 16

l = -9 → 9

6 standard reflections

monitored every 150

reflections

intensity variation: 3%

Refinement

Refinement on *F*²

Final *R* = 0.069

wR = 0.076

S = 1.84

5487 reflections

337 parameters

w = [σ_c²(*F_o*²) + (0.030*F_o*²)²]⁻¹

(Δ/σ)_{max} = 0.006

Δρ_{max} = 2.0 e Å⁻³

Δρ_{min} = -0.82 e Å⁻³

Atomic scattering factors

from *International Tables*

for X-ray Crystallography

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²)

Disordered F atoms have occupancies: A 0.443(16); B 0.557(16).

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
As	0.05093 (4)	0.24527 (4)	0.53284 (7)	0.0475 (3)
S1	0.58603 (8)	0.24910 (8)	0.34756 (15)	0.0438 (6)
S2	0.32644 (8)	0.11471 (8)	0.16685 (15)	0.0437 (6)
S3	0.26833 (8)	0.77845 (8)	0.07563 (17)	0.0491 (6)
S4	0.53728 (8)	0.91124 (8)	0.18947 (15)	0.0443 (6)
F1A	-0.014 (2)	0.133 (2)	0.335 (3)	0.120 (12)
F1B	-0.0380 (16)	0.1603 (18)	0.325 (2)	0.137 (10)
F2A	0.1186 (16)	0.3573 (14)	0.731 (2)	0.107 (10)
F2B	0.1424 (16)	0.3332 (16)	0.741 (2)	0.166 (11)
F3A	0.1807 (11)	0.300 (2)	0.475 (3)	0.132 (12)
F3B	0.1405 (13)	0.3335 (10)	0.3943 (18)	0.107 (8)

F4A	-0.0802 (10)	0.1951 (18)	0.597 (2)	0.138 (12)
F4B	-0.0390 (13)	0.1567 (10)	0.662 (2)	0.131 (9)
F5A	0.0122 (12)	0.3668 (9)	0.454 (2)	0.131 (9)
F5B	-0.0219 (14)	0.3272 (16)	0.517 (2)	0.219 (15)
F6A	0.0911 (16)	0.1320 (11)	0.632 (2)	0.140 (10)
F6B	0.1273 (14)	0.1621 (15)	0.539 (3)	0.191 (14)
C1	0.5626 (3)	0.4755 (3)	0.2855 (5)	0.034 (2)
C2	0.5106 (3)	0.3443 (3)	0.2694 (5)	0.032 (2)
C3	0.3858 (3)	0.2798 (3)	0.1861 (5)	0.032 (2)
C4	0.3177 (3)	0.3488 (3)	0.1275 (5)	0.036 (2)
C5	0.3008 (3)	0.5538 (3)	0.0959 (5)	0.035 (2)
C6	0.3505 (3)	0.6841 (3)	0.1178 (5)	0.032 (2)
C7	0.4784 (3)	0.7485 (3)	0.1863 (5)	0.029 (2)
C8	0.5466 (3)	0.6798 (3)	0.2410 (5)	0.034 (2)
C9	0.4940 (3)	0.5461 (3)	0.2249 (5)	0.030 (2)
C10	0.3702 (3)	0.4822 (3)	0.1473 (5)	0.031 (2)
C11	0.7374 (4)	0.3603 (4)	0.4280 (7)	0.049 (3)
C12	0.1697 (4)	0.0638 (4)	0.0777 (8)	0.058 (3)
C13	0.1169 (4)	0.6645 (4)	0.0154 (8)	0.057 (3)
C14	0.6861 (4)	0.9708 (4)	0.3326 (7)	0.054 (3)

Table 2. Geometric parameters (Å, °)

As—F1A	1.68 (2)	S3—C6	1.759 (4)
As—F1B	1.65 (1)	S3—C13	1.792 (4)
As—F2A	1.68 (1)	S4—C7	1.724 (3)
As—F2B	1.67 (1)	S4—C14	1.791 (5)
As—F3A	1.64 (1)	C1—C2	1.381 (5)
As—F3B	1.71 (1)	C1—C9	1.421 (6)
As—F4A	1.67 (1)	C2—C3	1.433 (4)
As—F4B	1.67 (1)	C3—C4	1.398 (6)
As—F5A	1.74 (1)	C4—C10	1.402 (5)
As—F5B	1.53 (2)	C5—C6	1.366 (5)
As—F6A	1.72 (2)	C5—C10	1.425 (6)
As—F6B	1.58 (2)	C6—C7	1.450 (4)
S1—C2	1.754 (4)	C7—C8	1.391 (6)
S1—C11	1.788 (4)	C8—C9	1.407 (5)
S2—C3	1.736 (3)	C9—C10	1.416 (4)
S2—C12	1.789 (5)		
C2—S1—C11	103.7 (2)	S3—C6—C5	124.0 (3)
C3—S2—C12	105.7 (2)	S3—C6—C7	118.0 (2)
C6—S3—C13	103.6 (2)	C5—C6—C7	117.9 (4)
C7—S4—C14	106.3 (2)	S4—C7—C6	115.7 (3)
C2—C1—C9	121.1 (3)	S4—C7—C8	123.7 (2)
S1—C2—C1	124.7 (3)	C6—C7—C8	120.6 (3)
S1—C2—C3	116.8 (3)	C7—C8—C9	121.0 (3)
C1—C2—C3	118.5 (4)	C1—C9—C8	121.3 (3)
S2—C3—C2	116.2 (3)	C1—C9—C10	120.2 (3)
S2—C3—C4	123.2 (2)	C8—C9—C10	118.5 (4)
C2—C3—C4	120.6 (3)	C4—C10—C5	121.3 (3)
C3—C4—C10	120.8 (3)	C4—C10—C9	118.7 (4)
C6—C5—C10	121.9 (3)	C5—C10—C9	119.9 (3)

Table 3. Shortest intermolecular contact distances (S...S ≤ 3.7 Å and H...F ≤ 2.5 Å) and angles (°)

	S...S/H...F	C—H...F	No. in Fig. 1
S2...S3 ⁱ	3.625 (2)	—	Interstack
S2...S4 ^{iv}	3.338 (2)	—	Intrastack
S4...S4 ⁱⁱ	3.701 (2)	—	Interstack
C1—H1...F3A ^v	2.32 (3)	151 (3)	AsF ₆ ⁻
C4—H2...F2B ⁱⁱ	2.48 (3)	118 (2)	AsF ₆ ⁻
C5—H3...F2A ⁱⁱⁱ	2.44 (3)	124 (2)	AsF ₆ ⁻
C11—H5...F5B ⁱⁱ	2.21 (4)	141 (3)	AsF ₆ ⁻
C11—H7...F3B ^v	2.50 (3)	129 (3)	AsF ₆ ⁻
C12—H8...F1A	2.41 (6)	158 (3)	AsF ₆ ⁻
C12—H9...F6A ⁱⁱ	2.32 (5)	171 (3)	AsF ₆ ⁻
C14—H15...F6A ^v	2.44 (5)	145 (2)	AsF ₆ ⁻
C14—H15...F6B ^v	2.41 (5)	136 (3)	AsF ₆ ⁻

Symmetry code: (none) *x*, *y*, *z*; (i) *x*, *y* - 1, *z*; (ii) *x*, *y*, *z* - 1; (iii) *x* + 1, *y*, *z*; (iv) 1 - *x*, 1 - *y*, -*z*; (v) 1 - *x*, 1 - *y*, 1 - *z*; (vi) 1 - *x*, 2 - *y*, -*z*.

Crystals were prepared by electrolytic oxidation of TMTN in CH₂Cl₂ solution in the presence of (*n*-Bu₄N)AsF₆ at a constant current density of a few μA cm⁻² at room temperature. The

number of scans was varied to maintain constant $\sigma(F)/F = 0.017$ with a maximum of eight scans; ω -scan rate 8° min^{-1} with ω -scan width $(0.787 + 0.3 \tan \theta)^\circ$. The crystal chosen for experiment was found to be a twin crystal, where the $hk0$ reflections from the two crystal halves overlap each other. The data were collected for the halves as separate subsets. A value of R_{int} is missing since in each subset only a unique data set was collected. The data files were merged without averaging. From the diffraction symmetry the space groups $P1$ and $\bar{P}1$ were possible. The structure was solved and refined in $P1$; lowering the symmetry to $\bar{P}1$ gave no significant improvement in the R factors. The As and S atoms were found in a Patterson synthesis; the rest of the structure (except for H atoms) was found in successive Fourier syntheses. All H-atom positions were first calculated geometrically, using a C—H distance of 1.08 Å. These positions were subsequently refined without constraints. The C-ring H atoms were restricted to have identical isotropic displacement parameters. Similarly, the displacement parameters of the H atoms in each —CH₃ group were kept the same. The two subsets were given different scale factors in the refinement. For the $hk0$ reflections two other scale factors were introduced, making in total four different scale factors to be used in the refinements. After the final refinement all H-atom positions were recalculated (C—H 1.08 Å). In the geometrical calculations (*cf.* Table 3) e.s.d.'s for the H-atom positions were taken from the refinement. The molecular plots were made using *ORTEPII* (Johnson, 1976). Other calculations were made using the *TEXSAN* program system (Molecular Structure Corporation, 1989).

This work has been supported by a grant from the National Swedish Board for Technical Development, which is hereby gratefully acknowledged. The authors also wish to thank Dr Jonas Hellberg for providing the crystals and Hilding Karlsson for expert technical advice during the data collection.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55473 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1022]

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Acta Cryst. (1993). **C49**, 171–173

Structure of Kopsine: Methyl 3-Hydroxy-22-oxokopsan-1-carboxylate

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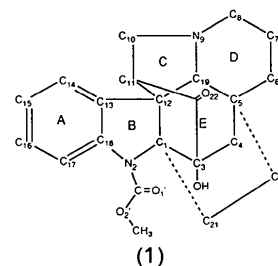
(Received 30 July 1992; accepted 24 September 1992)

Abstract

The Kopsine molecule consists of five fused rings. The phenyl ring *A* is almost planar. The conformation of ring *B* is a pure envelope, ring *C* is a half-chair, ring *D* is a chair and ring *E* is a boat. An intramolecular hydrogen bond $\text{O}(3) \cdots \text{H}(\text{O}3) \cdots \text{O}(1')$ helps to stabilize the boat conformation of ring *E*.

Comment

Kopsine, $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_4$, m.p. 487–489 K, was isolated from the leaves of *Kopsia fruticosa*. The structure of Kopsine (1) with its absolute configuration was previously deduced by Guggisberg, Gorman, Bycroft & Schmid (1969) through the application of physical methods, mainly NMR. The complete structure of Kopsine with relative configuration and various ring conformations has been established by X-ray crystallography.



The isolation was carried out as follows: air dried and coarsely powdered leaves (750 g) of *Kopsia fruticosa* (identified by Dr S. R. Das, Plant Survey Officer, CCRAS, Calcutta, and a specimen preserved in the laboratory) were extracted with petroleum ether (b.p. 333–353 K) in a soxhlet apparatus for 18 h. The concentrated