Fig. 2. Unit-cell packing diagrams with hydrogen bonds indicated by thin lines. There are no interbase hydrogen bonds. Hydrogens attached to carbon atoms have been omitted for clarity. (a) Projection along the b axis illustrating the base stacking along the twofold screw axis parallel to the a axis. The bases are essentially perpendicular to the a axis and stacked about 3·4 Å apart. (b) Projection along the a axis showing the flip-flop overlap of bases in the stacks and the alignment of the amino bond with the N4—C9 bond.

Related literature. The title compound is related to formycin A first isolated by Hori, Ito, Takita, Koyama, Takeuchi & Umezawa (1964). Structures of formycin and derivatives include formycin monohydrate (Prusiner, Brennan & Sundaralingam, 1973), 2-methylformycin (Abola, Sims, Abraham, Lewis & Townsend, 1974) and formycin 5'-phosphate (Giranda, Berman & Schramm, 1988). Comparison of (1) with the structure of the unsubstituted 1,2,4-triazolo[4,3-b]pyridazine

reported by Golic, Leban, Stanovnik & Tisler (1978) indicates a slight bond elongation occurring at bonds involving the substitution sites, C3 and C8. The conformational parameters follow the convention of Altona & Sundaralingam (1972). The title compound was found to lack significant biological activity (Kang, Larson, Robins & Revankar, 1989).

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1-Phenyl-N²-(1-phenylmethyl-3-isoquinolyl)acetamidinium Trifluoromethanesulfonate

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Abstract. $C_{24}H_{22}N_3^+.CF_3O_3S^-$, $M_r = 501\cdot5$, monoclinic, $P2_1/c$, $a = 7\cdot022$ (1), $b = 18\cdot294$ (3), $c = 18\cdot617$ (4) Å, $\beta = 91\cdot33$ (2)°, $V = 2390\cdot9$ Å³, $Z = 4$, $D_x = 1\cdot392$ Mg m⁻³, $F(000) = 1040$, $\lambda(Mo\ K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot148$ mm⁻¹, $T = 293$ K, $R = 0\cdot041$ for 2260 unique reflexions $[F \geq 3\sigma(F)]$. Coplanar acetamidinium and isoquinoline moieties $[C—C—N—C = -179\cdot3$ (1)°] are able to interact through their π systems. The protonated =NH group takes part in two hydrogen bonds: one is a weak intramolecular interaction with the isoquinoline nitrogen $[=NH\cdots N = 1\cdot98$ (3), $N\cdots N = 2\cdot658$ (5) Å, $N—H\cdots N = 135$ (2)°] and the other is to an oxygen O(33) of the anion $[=NH\cdots O(33) = 2\cdot02$ (3) Å, $N\cdots O(33) = 2\cdot886$ (5) Å, $N—H\cdots O(33) = 171$ (2)°]. The second acetamidinium nitrogen also forms an H bond to

$C_{24}H_{22}N_3^+.CF_3O_3S^-$

Table 1. *Final coordinates* ($\times 10^5$ *for* S; $\times 10^4$ *for* C, N, O, F) *and vibrational parameters* ($Å^2 \times 10^4$) *for non-hydrogen atoms*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	2562 (4)	3907 (2)	5312 (2)	496 (8)
N(2)	2115 (3)	3857 (1)	4620 (1)	468 (6)
C(3)	1952 (4)	4480 (1)	4230 (1)	429 (7)
C(4)	2206 (4)	5161 (1)	4506 (2)	490 (8)
C(5)	2983 (4)	5910 (2)	5578 (2)	668 (10)
C(6)	3459 (5)	5936 (2)	6294 (2)	797 (11)
C(7)	3620 (5)	5305 (2)	6705 (2)	768 (11)
C(8)	3321 (4)	4647 (2)	6398 (2)	658 (9)
C(9)	2846 (4)	4585 (2)	5662 (2)	503 (8)
C(10)	2668 (4)	5223 (2)	5244 (2)	495 (8)
C(11)	2801 (5)	3187 (2)	5710 (2)	671 (9)
N(12)	1490 (3)	4388 (1)	3495 (1)	481 (6)
C(13)	1170 (4)	3778 (2)	3125 (2)	497 (7)
N(14)	1261 (4)	3135 (1)	3416 (1)	620 (7)
C(15)	751 (4)	3847 (2)	2336 (2)	610 (8)
C(16)	2533 (4)	3955 (2)	1909 (2)	575 (8)
C(17)	3922 (5)	3425 (2)	1898 (2)	716 (9)
C(18)	5551 (6)	3518 (3)	1526 (2)	955 (13)
C(19)	5820 (7)	4142 (3)	1151 (3)	1114 (15)
C(20)	4468 (9)	4674 (3)	1141 (3)	1244 (18)
C(21)	2796 (6)	4578 (2)	1523 (2)	886 (12)
C(22)	2270 (5)	2525 (2)	5285 (2)	603 (9)
C(23)	3571 (5)	2187 (2)	4850 (2)	835 (11)
C(24)	3047 (7)	1585 (2)	4445 (2)	1047 (15)
C(25)	1263 (9)	1310 (2)	4477 (2)	1076 (16)
C(26)	−26 (6)	1638 (3)	4905 (2)	1011 (13)
C(27)	463 (5)	2245 (2)	5308 (2)	753 (10)
C(28)	6930 (6)	3328 (2)	7866 (2)	772 (11)
F(29)	7428 (3)	3555 (1)	8511 (1)	1095 (8)
F(30)	6611 (4)	2632 (1)	7931 (2)	1504 (11)
F(31)	5360 (4)	3652 (2)	7703 (1)	1490 (12)
O(32)	9078 (3)	4267 (1)	7275 (1)	782 (7)
O(33)	10317 (4)	3066 (1)	7467 (1)	1039 (8)
O(34)	7885 (4)	3278 (1)	6548 (1)	1047 (9)
S(1)	87542 (13)	35036 (4)	72164 (5)	685 (2)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

C(1)–N(2)	1·323 (4)	C(1)–C(9)	1·412 (4)
C(1)–C(11)	1·520 (4)	N(2)–C(3)	1·354 (3)
C(3)–C(4)	1·357 (4)	C(3)–N(12)	1·410 (3)
C(4)–C(10)	1·409 (4)	C(5)–C(6)	1·368 (5)
C(5)–C(10)	1·418 (4)	C(6)–C(7)	1·389 (6)
C(7)–C(8)	1·346 (5)	C(8)–C(9)	1·408 (4)
C(9)–C(10)	1·407 (4)	C(11)–C(22)	1·489 (4)
N(12)–C(13)	1·328 (3)	C(13)–N(14)	1·296 (4)
C(13)–C(15)	1·497 (4)	C(15)–C(16)	1·511 (4)
C(16)–C(17)	1·375 (5)	C(16)–C(21)	1·363 (4)
C(17)–C(18)	1·362 (6)	C(18)–C(19)	1·353 (7)
C(19)–C(20)	1·360 (8)	C(20)–C(21)	1·397 (7)
C(22)–C(23)	1·381 (5)	C(22)–C(27)	1·370 (5)
C(23)–C(24)	1·380 (6)	C(24)–C(25)	1·353 (8)
C(25)–C(26)	1·358 (7)	C(26)–C(27)	1·380 (6)
C(28)–F(29)	1·310 (4)	C(28)–F(30)	1·298 (4)
C(28)–F(31)	1·282 (5)	C(28)–S(1)	1·810 (4)
O(32)–S(1)	1·419 (2)	O(33)–S(1)	1·428 (3)
O(34)–S(1)	1·434 (3)		

N(2)–C(1)–C(9)	122·6 (2)	N(2)–C(1)–C(11)	115·8 (2)
C(9)–C(1)–C(11)	121·6 (3)	C(1)–N(2)–C(3)	118·5 (2)
N(2)–C(3)–C(4)	124·2 (2)	N(2)–C(3)–N(12)	115·7 (2)
N(12)–C(13)–N(14)	122·8 (3)	N(12)–C(13)–C(15)	117·7 (2)
N(14)–C(13)–C(15)	119·6 (2)	C(3)–N(12)–C(13)	129·4 (2)

O(32) of a separate anion [>NH···O(32) = 2·04 (2), N···O(32) = 2·870 (4) Å, N–H···O(32) = 175 (2)°], thus forming chains parallel to **b**.

Experimental. The reaction between phenylacetonitrile and triflic acid (1:1 mole ratio) at room temperature over 42 h gave yellow crystals suitable for X-ray work.

Crystal dimensions 0·2 × 0·2 × 0·3 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochro-

mated Mo Kα radiation, unit-cell dimensions from setting angles of 25 accurately centred reflexions ($11·5 \le \theta \le 14·6°$), ω–2θ scan mode, ω scan width ($0·70 + 0·35\tan\theta$)° and scan speed ranging from 0·3 to 5° min^{-1} according to the intensity gathered in a pre-scan, $-6 \le h \le 6$, $0 \le k \le 17$, $0 \le l \le 20$, $0 \le \theta \le 25°$, 3701 reflexions measured, 2705 unique (R_{int} = 0·015), 2260 observed [$F \ge 3\sigma(F)$], intensity standards ($\overline{2}29$, $\overline{1}6\overline{8}$, $\overline{2}8\overline{4}$) measured every 2 h, no systematic drift, Lp corrections applied but absorption ignored, MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) used to solve the phase problem, all non-hydrogen atoms found in Fourier map, hydrogen atoms from ΔF synthesis, blocked-matrix least squares based on F using SHELX76 (Sheldrick, 1976), final $R = 0·041$ {wR = 0·048, $w = 0·6024/[\sigma^2(F) + 0·0002F^2]$}, anisotropic thermal parameters for heavier atoms, isotropic for hydrogen. Maximum fluctuation in final ΔF map in range $-0·23$ to $0·19$ e Å^{-3}, maximum $\Delta/\sigma = 0·1$. Scattering factors from *International Tables for X-ray Crystallography* (1974), computation carried out on the joint CDC 7600/Amdahl 470 system of the University of Manchester Regional Computing Centre. Literature survey performed *via* the Cambridge Structural Database using the Crystal Structure Search and Retrieval interactive system (CSSR, 1984). Fractional atomic coordinates and vibrational parameters for non-hydrogen atoms are presented in Table 1* and selected bond lengths and angles in Table 2. The cation

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* Lists of structure factors, hydrogen coordinates, distances and angles involving H atoms and anisotropic vibrational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51733 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
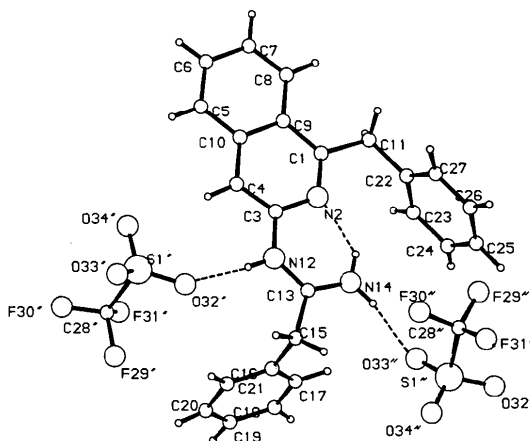

Fig. 1. The title molecular ions including the hydrogen-bonding scheme drawn using PLUTO (Motherwell & Clegg, 1978). Single and double primes denote atoms generated by 1−x, 1−y, 1−z and x−1, $\frac{1}{2}$−y, z−$\frac{1}{2}$, respectively.

and anion, including atomic labeling, are displayed in Fig. 1.

Related literature. A full discussion of the method of preparation is given by Booth & Collis (1988). Earlier work by Amer, Booth, Noori & Proença (1983) and Norell (1976) suggested that the product would be 2,4,6-tribenzyl-1,3,5-triazine and not the title molecule, whose structure was established as a result of this X-ray investigation. The isoquinoline nucleus resembles those in several reported structures, *e.g.* McClure & Schlemper (1978), Ljungström & Lindqvist (1978), and Ribár, Divjaković, Janić, Argay, Kálmán & Djurić (1974).

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Structure of Thiolactomycin δ

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Abstract. [4RS,5SR-(2E,6E)]-3-Hydroxy-2,4-dimethyl-5-(1-methyl-1-propenyl)-2-penten-5-thiolide,† $C_{11}H_{16}O_2S$, $M_r = 212\cdot31$, triclinic, $P\bar{1}$, $a = 8\cdot6196$ (6), $b = 10\cdot387$ (1), $c = 6\cdot9962$ (4) Å, $\alpha = 95\cdot97$ (1), $\beta = 99\cdot40$ (1), $\gamma = 107\cdot22$ (1)°, $V = 582\cdot5$ Å³, $Z = 2$, $D_x = 1\cdot210$ g cm⁻³, $\lambda(Cu\,K\alpha) = 1\cdot5418$ Å, $\mu = 22\cdot14$ cm⁻¹, $F(000) = 228$, $T = 298$ K, final $R = 0\cdot059$ for 1988 unique reflections [$F_o^2 > 2\sigma(F_o^2)$]. The title compound is a racemate and one of the homologues of thiolactomycin. The thiolactone ring adopts a sofa conformation, with S—C bond distances of 1·750 (4) and 1·824 (3) Å, and a C—S—C angle of 101·8 (2)°. The angle between the least-squares planes of the five ring atoms and the propenyl moiety is 144·6°. Intermolecular hydrogen bonding and van der Waals contacts are observed: O(6)···O(8) (x, y, 1+z) = 2·581 (3) and C(7)···C(7) (1−x, 1−y, 1−z) = 3·633 (6) Å.

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† Alternative name: [4RS,5SR-(2E,6E)]-3-hydroxy-2,4-dimethyl-5-(1-methyl-1-propenyl)-6-thiacyclohex-2-en-1-one.

Experimental. The title compound was isolated from the culture broth (Oishi, Noto, Sasaki, Suzuki, Hayashi. Okazaki, Ando & Sawada, 1982) by silica-gel column chromatography (eluent: benzene), prior to the elution of thiolactomycin (eluent: benzene/acetone = 95:5, v/v). Colorless prisms were grown from acetone solution (m.p. 403 K; found: C 62·4, H 7·8, S 15·0%. $C_{11}H_{16}O_2S$ requires C 62·2, H 7·6, S 15·1%). Crystal size 0·38 × 0·25 × 0·18 mm, Enraf–Nonius CAD-4 κ-cradle diffractometer, Cu $K\alpha$ radiation, graphite monochromator, θ–2θ scan with scan speed 2·75–5·49° min⁻¹ in θ, scan width (0·8 + 0·2tanθ)°. Range of indices, $-10 \le h \le 10$, $-13 \le k \le 13$, $0 \le l \le 8$ (2θ < 150°). Lattice constants determined based on 25 2θ values (21 < θ < 35°). Variation of standard < 1·3%; 2382 unique reflections measured; 1988 observed reflections with $F_o^2 > 2\sigma(F_o^2)$. No correction for absorption. Structure solved by direct methods with *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Refined by full-matrix least squares. The locations of all the H atoms were found on difference Fourier maps. Non-H