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Synthesis of 2-Thioxo-4,6-diphenyl-1,2-dihydronicotinonitrile via Condensation of Benzaldehyde with Cyanothioacetamide and *p*-(1-Styryl)morpholine

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Abstract—2-Thioxo-4,6-diphenyl-1,2-dihydronicotinonitrile has been prepared via condensation of benzaldehyde with cyanothioacetamide and *N*-(1-styryl)morpholine; alkylation of the product with alkyl halides has afforded substituted 2-alkylsulfanyl-4,6-diphenylnicotinonitriles and 3-amino-2-acyl-4,6-diphenylthieno[2,3-*b*]pyridines.

Keywords: benzaldehyde, cyanothioacetamide, *N*-(1-styryl)morpholine, 2-thioxodihydronicotinonitrile, 2-alkylsulfanylnicotinonitrile, thieno[2,3-*b*]pyridine

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Derivatives of aryl(hetaryl)-substituted 2-oxo(thioxo)-1,2-dihydronicotinonitrile are biologically active compounds exhibiting antimicrobial [1, 2], antitumor [3], and herbicide [4] activity. They can be prepared via interaction of δ-ketodinitriles with elemental sulfur [5, 6] or hydrogen sulfide [7], chalcones reaction with cyanothioacetamide [7, 8], and by condensation of aryl-(hetaryl)methylidenecyanothioacetamides with acetophenones [9–11] or their enamines [11].

Extending the studies on the chemistry of 4,6-diaryl-2-thioxo-1,2-dihydronicotinic acid derivatives [12–14], we developed a new procedure for the preparation of such organic compounds based on the condensation of benzaldehyde (**I**) with cyanothioacetamide (**II**) and *N*-(1-styryl)morpholine (**III**) in anhydrous ethanol at 20°C; the reaction product was 2-thioxo-4,6-diphenyl-1,2-dihydronicotinonitrile (**IV**). The reaction likely involved the formation of Knoevenagel alkene (**A**) that alkylated the enamine **III** via the Stork reaction [15] to yield the adduct **B**. The latter underwent the intramolecular transamination [16] followed by dehydration (probably, aided by air oxygen) to form compound **IV**, earlier prepared via the reaction of δ-ketodinitrile with elemental sulfur [5] (Scheme 1).

The alkylation of compound **IV** with alkyl halides **Va**–**Vm** in DMF solution in the alkali medium occurred regioselectively at the sulfur atom to yield the thioesters **VIa**–**VIm** capable of the intramolecular cyclization at their treatment with KOH solution in aqueous DMF affording the 2-alkoxycarbonyl-3-amino-4,6-diphenylthieno[2,3-*b*]pyridines **VIIa**–**VIIc**, potential intermediate products for the synthesis of antitumor [17, 18] and antiviral [19] drugs.

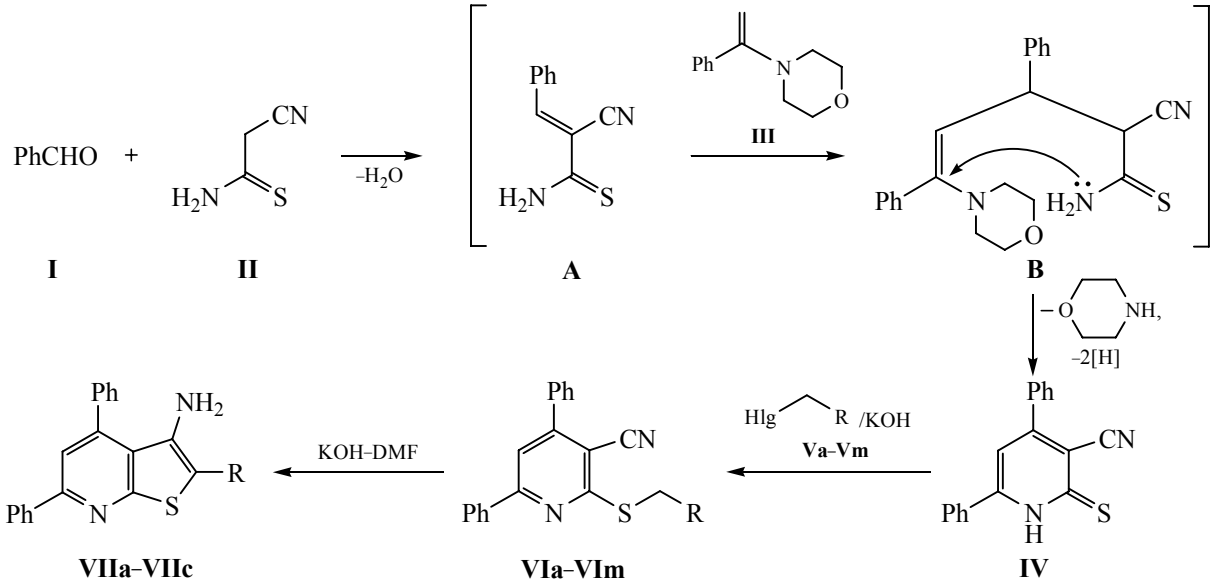
Structure of the prepared compounds **VIa**–**VIm** and **VIIa**–**VIIc** was confirmed by their physicochemical and spectral properties (Tables 1 and 2).

In particular, IR spectra of compounds **VI** contained characteristic absorption bands of the conjugated nitrile group stretching vibrations at 2214–2226 cm^{-1}. Those signals were absent in the spectra of the thienopyridines **VII**; instead, the bands assigned to stretching and bending vibrations of the amino group were observed at 3198–3350 and 1638–1645 cm^{-1}, respectively.

^1H NMR spectra of the thioesters **VI** were remarkable for the presence of a singlet signal of the H^5 proton of pyrimidine ring at 7.38–7.85 ppm and that of the SCH_2 group at 4.11–5.11 ppm; those signals disappeared after the substrates transformation into the thienopyridines **VII**. ^1H NMR spectra of compounds **VII** contained the broadened singlet signal of the amino group protons at 5.80–6.51 ppm.

Mass spectra of compounds **VIb**, **VIi**–**VIm** contained weak signals of the [*M* + 2] ion peaks, pointing at the presence of a single S atom in the

Scheme 1.



V–VII, Hlg=Br, R=Me_2CHCH_2CO (**a**); Cl, Me_2CHOCO (**b**); Br, $Me(CH_2)_3CO$ (**c**); Br, 4-cyclohexylbenzoyl (**d**); Br, HC≡C (**e**); Br, PhCO (**f**); Cl, MeOCO (**g**); Cl, quinolin-8-ylcarbamoyl (**h**); Cl, $PhCH_2OCO$ (**i**); Cl, PhNHCO (**j**); Cl, 4-AcC_6H_4NHCO (**k**); Cl, $Me(CH_2)_7OCO$ (**l**); Cl, $Me(CH_2)_8OCO$ (**m**).

Table 1. Yields, melting points, and elemental analysis data for 2-alkylsulfanyl-4,6-diphenylnicotinonitriles (**VIa–VIm**) and 3-amino-2-acyl-4,6-diphenylthieno[2,3-*b*]pyridines (**VIIa–VIIc**)

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
VIa	82	105–106 (PrOH)	74.42	5.66	7.11	$C_{24}H_{22}N_2OS$	74.58	5.74	7.25
VIb	70	165–166 (AcOH)	71.01	5.04	7.12	$C_{23}H_{20}N_2O_2S$	71.11	5.19	7.21
VIc	74	110–112 (AcOH)	74.49	5.68	7.09	$C_{24}H_{22}N_2OS$	74.58	5.74	7.25
VId	85	176–178 (AcOH)	78.50	5.61	5.68	$C_{32}H_{28}N_2OS$	78.66	5.78	5.73
VIe	70	132–134 (EtOH)	77.14	4.25	8.41	$C_{21}H_{14}N_2S$	77.27	4.32	8.58
VIf	81	182–184 (AcOH)	76.70	4.39	6.71	$C_{26}H_{18}N_2OS$	76.82	4.46	6.89
VIg	76	191–193 (MeOH)	69.83	4.32	7.60	$C_{21}H_{16}N_2O_2S$	69.98	4.48	7.77
VIh	77	211–213 (BuOH)	73.62	4.15	11.73	$C_{29}H_{20}N_4OS$	73.71	4.27	11.86
VIi	74	140–141 (EtOH)	74.08	4.50	6.32	$C_{27}H_{20}N_2O_2S$	74.29	4.62	6.42
VIj	82	218–220 (BuOH)	73.95	4.41	9.80	$C_{26}H_{19}N_3OS$	74.09	4.54	9.97
VIk	76	225–227[a] (BuOH)	72.44	4.64	8.95	$C_{28}H_{21}N_3O_2S$	72.55	4.57	9.07
VIl	75	98–99 (MeOH)	73.18	6.44	5.97	$C_{28}H_{30}N_2O_2S$	73.33	6.59	6.11
VIm	69	96–97 (MeOH)	73.54	6.70	5.86	$C_{29}H_{32}N_2O_2S$	73.69	6.82	5.93
VIIa	79	100–102 (BuOH)	74.49	5.64	7.17	$C_{24}H_{22}N_2OS$	74.58	5.74	7.25
VIIb	68	115–116 (*i*-PrOH)	70.96	5.10	7.12	$C_{23}H_{20}N_2O_2S$	71.11	5.19	7.21
VIIc	70	80–82 (MeOH)	74.40	5.64	7.15	$C_{24}H_{22}N_2OS$	74.58	5.74	7.25

[a] Sublimates at 160°C.

Table 2. IR and ^{1}H NMR spectral data for 2-alkylsulfanyl-4,6-diphenylnicotinonitriles (**VIa**–**VIm**) and 3-amino-2-acyl-4,6-diphenylthieno[2,3-*b*]pyridines (**VIIa**–**VIIc**)

Comp. no.	ν, cm^{-1}		δ, ppm (*J*, Hz)	
	C≡N or NH_2	C=O, δ(NH_2)	H^5, SCH_2, or NH_2	other signals
VIa	2218	1688	7.46, 4.18	1.91 d (6H, 2Me, *J* 5.1), 2.03–2.15 m (1H, CHMe$_2$), 2.54 d (2H, CH_2CO, *J* 6.0), 7.52–7.61 m (4H, Ph), 7.65–7.73 m (4H, Ph), 8.05 d (2H, Ph, *J* 7.9)
VIb	2215	1719	7.85, 4.17	1.18 d (6H, 2Me, *J* 4.8), 4.94 q (1H, CHMe$_2$, *J* 4.8), 7.38–7.72 m (8H, H_{arom}), 8.11–8.26 m (2H, Ph)
VIc	2219	1713	7.38, 4.19	0.85 t (3H, Me, *J* 7.5), 1.18–1.26 m (2H, CH_2), 1.43–1.59 m (2H, CH_2), 2.65 t (2H, CH_2CO, *J* 7.5), 7.44–7.58 m (6H, H_{arom}), 7.82–7.87 m (2H, Ph), 8.04 d (2H, Ph, *J* 7.5)
VId	2220	1715	7.79, 4.92	1.21–1.62 m (5H, cyclohexane), 1.73–1.99 m (5H, cyclohexane), 2.68 t (1H, cyclohexane, *J* 5.4), 7.21 t (2H, Ph, *J* 6.9), 7.39 t (3H, Ph, *J* 6.9), 7.55 t (3H, Ph, *J* 7.1), 7.73 d (2H, Ph, *J* 7.1), 7.90 d (2H, C_6H_4, *J* 7.6), 8.05 d (2H, C_6H_4, *J* 7.6)
VIe	2212	2244 (C≡C)	7.83, 4.21	2.90 s (1H, ≡CH), 7.48–7.63 m (6H, 2Ph), 7.71 d (2H, Ph, *J* 6.9), 8.29 d (2H, Ph, *J* 7.1)
VIf	2223	1700	7.69, 4.93	7.12 t (2H, Ph, *J* 7.7), 7.33 t (1H, Ph, *J* 6.4), 7.43–7.58 m (4H, Ph), 7.60–7.78 m (4H, Ph), 7.83 d (2H, Ph, *J* 8.0), 8.09 d (2H, Ph, *J* 8.0)
VIg	2218	1720	7.80, 4.18	3.78 s (3H, Me), 7.38–7.61 m (6H, 2Ph), 7.68 d (2H, Ph, *J* 7.1), 8.14 d (2H, Ph, *J* 6.9)
VIh	2221	1666	7.82, 4.44	7.16–7.65 m (10H, H_{arom}), 8.16–8.39 m (4H, H_{arom}), 8.51–8.72 m (2H, H_{arom}), 10.70 br.s (1H, NH)
VIi	2225	1714	7.76, 5.11	4.24 s (2H, OCH_2), 7.26 br.s (5H, Ph), 7.31–7.64 m (8H, Ph), 8.13 d (2H, Ph, *J* 7.1)
VIj	2220	1670	7.72, 4.24	7.00 t (1H, Ph, *J* 7.1), 7.09–7.33 m (5H, Ph), 7.48 br.s (5H, Ph), 7.64 d (2H, Ph, *J* 6.9), 8.19 d (2H, Ph, *J* 6.9), 10.23 br.s (1H, NH)
VIk	2222	1663	7.75[a], 4.29	2.54 s (3H, Me), 7.26–7.51 m (3H, H_{arom}), 7.57–7.90[a] m (8H, H_{arom}), 7.96 t (2H, H_{arom}, *J* 7.1), 8.13 d (2H, H_{arom}, *J* 6.9), 10.60 br.s (1H, NH)
VIl	2226	1718	7.74, 4.11	0.82 t (3H, Me, *J* 6.7), 0.92–1.31 br.s (10H, $5CH_2$), 1.39–1.64 m (2H, CH_2), 4.01 t (2H, OCH_2, *J* 6.8), 7.31–7.66 m (8H, H_{arom}), 8.02–8.19 m (2H, Ph)
VIm	2225	1715	7.73, 4.13	0.85 t (3H, Me, *J* 5.6), 1.01–1.39 br.s (12H, $6CH_2$), 1.43–1.70 m (2H, CH_2), 4.06 t (2H, OCH_2, *J* 6.2), 7.41–7.67 m (8H, H_{arom}), 8.02–8.20 m (2H, H_{arom})
VIIa	3198, 3270, 3331	1690, 1638	7.78, 6.45	0.94 d (6H, 2Me, *J* 5.2), 2.11–2.22 m (1H, CHMe$_2$), 2.54 d (2H, CH_2CO, *J* 5.1), 7.41–7.46 m (2H, Ph), 7.49–7.66 m (6H, H_{arom}), 8.22 d (2H, Ph, *J* 8.2)
VIIb	3205, 3272, 3345	1699, 1645	7.80, 5.80	1.30 d (6H, 2Me, *J* 5.1), 5.06–5.19 m (1H, CHMe$_2$), 7.48–7.53 m (3H, Ph), 7.62 br.s (5H, Ph), 8.23 d (2H, Ph, *J* 7.1)
VIIc	3200, 3291, 3350	1710, 1644	7.76, 6.51	0.88 t (3H, Me, *J* 5.6), 1.22–1.38 m (2H, CH_2), 1.59–1.63 m (2H, CH_2), 2.64 t (2H, OCH_2, *J* 5.5), 7.48 d (3H, Ph, *J* 7.5), 7.58 br.s (5H, Ph), 8.21 d (2H, Ph, *J* 7.6)

[a] Overlapping signals.

structures. The values of the molecular ion peaks coincided with the "nitrogen rule" [20].

To summarize, we have elaborated a new method of preparation of 2-thioxo-4,6-diphenyl-1,2-dihydronicotinonitriles based on the three-component condensation of benzaldehyde, cyanothioacetamide, and *N*-(1-styryl)morpholine.

EXPERIMENTAL

IR spectra of the KBr pellets were recorded using an UR-20 instrument. ^{1}H and ^{13}C NMR spectra of the solutions in DMSO-d_6 were registered using a Bruker 500 spectrometer (at 500.13 and 125.75 MHz, respectively) with TMS as the internal reference. Elemental analysis was performed using an EuroVector EA-3000 microanalyzer. Chromato-mass spectrometry analysis was performed using a Hewlett-Packard 5890/5972 GC-MS spectrometer (chemical ionization, HP5-MS column, solutions in CF_3COOH) for compounds **VIa**, **VIc–VIh** and **VIIa–VIIc** or a KRATOS 890A spectrometer (EI, 70 eV, direct injection of the substance in the ion source) for compounds **VIb, VIi–VIm**. Melting points were determined using a Koeffler heating block. The reactions course was monitored by TLC on the Silufol-254 plates (acetone–hexane 3 : 5, developing with iodine vapor or UV irradiation).

2-Thioxo-4,6-diphenyl-1,2-dihydronicotinonitrile (IV). A droplet of morpholine was added to a mixture of 1.0 mL (10 mmol) of benzaldehyde and 1.0 g (10 mmol) of cyanothioacetamide, and the mixture was stirred during 30 min. Enamine **III** [1.9 g (10 mmol) at 20°C] was then added, and the mixture was further stirred during 1 h. Then, after maintaining for 24 h the mixture was diluted with 10 wt % aqueous hydrochloric acid and further kept at room temperature for 2 h. The formed precipitate was filtered off and washed with water, ethanol, and hexane. Yield 2.3 g (79%), yellow crystals, mp 227–229°C (AcOH) (mp 228–230°C [5]).

2-Alkylsulfanyl-4,6-diphenylnicotinonitriles (VIa–VIm) *(general procedure)*. 5.6 mL of 10 wt % aqueous KOH (10 mmol of the alkali) and 10 mmol of the corresponding alkyl halide **Va–Vm** were added to a stirred solution of 2.9 g (10 mmol) of compound **IV** in 15 mL of DMF, and the mixture was stirred during 30 min. After keeping for 24 h the mixture was diluted with equal volume of water; the formed precipitate was filtered off and washed with water, ethanol, and hexane.

2-(2-Oxoisohexylsulfanyl)-4,6-diphenylnicotinonitrile (VIa). Mass spectrum, *m*/*z* (I_{rel}, %): 387 (100) $[M + 1]^+$. *M* 386.519. Fluorescent under UV irradiation.

Isopropyl-2-(4,6-diphenyl-3-cyanopyridin-2-ylsulfanyl) acetate (VIb). Mass spectrum, *m*/*z* (I_{rel}, %): 390 (3) $[M + 2]^+$, 389 (11) $[M + 1]^+$, 388 (33) $[M]^+$, 387 (39) $[M - 1]^+$, 345 (10), 329 (14), 301 (100) $[M - Me_3CHOCO]^+$, 255 (14), 227 (15), 140 (11), 77 (22) $[Ph]^+$, 44 (12) $[C{=}S]^+$, 43 (59) $[Me_2CH]^+$. *M* 388.491.

2-(2-Oxohexylsulfanyl)-4,6-diphenylnicotinonitrile (VIc). Mass spectrum, *m*/*z* (I_{rel}, %): 387 (100) $[M + 1]^+$. *M* 386.519. Fluorescent under UV irradiation.

4,6-Diphenyl-2-[2-oxo-2-(4-cyclohexylphenyl)-ethylsulfanyl]nicotinonitrile (VId). Mass spectrum, *m*/*z* (I_{rel}, %): 489 (100) $[M + 1]^+$. *M* 488.656.

2-Propargylsulfanyl-4,6-diphenylnicotinonitrile (VIe). Mass spectrum, *m*/*z* (I_{rel}, %): 327 (100) $[M + 1]^+$. *M* 326.423.

2-Benzoylmethylsulfanyl-4,6-diphenylnicotinonitrile (VIf). Mass spectrum, *m*/*z* (I_{rel}, %): 407 (100) $[M + 1]^+$. *M* 406.509.

Methyl-2-(4,6-diphenyl-3-cyanopyridin-2-yl) acetate (VIg). Mass spectrum, *m*/*z* (I_{rel}, %): 361 (100) $[M + 1]^+$. *M* 360.438.

***N*-(Quinolin-8-yl)-2-(4,6-diphenyl-3-cyanopyridin-2-ylsulfanyl) acetamide (VIh).** Mass spectrum, *m*/*z* (I_{rel}, %): 472 (100) $[M + 1]^+$. *M* 471.585.

Benzyl-2-(4,6-diphenyl-3-cyanopyridin-2-ylsulfanyl) acetate (VIi). Mass spectrum, *m*/*z* (I_{rel}, %): 438 (3) $[M + 2]^+$, 437 (9) $[M + 1]^+$, 436 (30) $[M]^+$, 435 (39) $[M - 1]^+$, 345 (5) $[M - PhCH_2]^+$, 301 (48), 288 (18), 255 (11), 227 (15), 91 (100) $[PhCH_2]^+$, 77 (21) $[Ph]^+$, 65 (12) 51 (5). *M* 436.536.

***N*-Phenyl-2-(4,6-diphenyl-3-cyanopyridin-2-ylsulfanyl) acetamide (VIj).** Mass spectrum, *m*/*z* (I_{rel}, %): 423 (4) $[M + 2]^+$, 422 (13) $[M + 1]^+$, 421 (45) $[M]^+$, 420 (5) $[M - 1]^+$, 329 (100) $[M - PhNH]^+$, 301 (56) $[M - PhNHCO]^+$, 255 (11), 211 (12), 93 (16) $[PhNH_2]^+$, 77 (15) $[Ph]^+$, 65 (7) 51 (5). *M* 421.524.

***N*-(4-Acetylphenyl)-2-(4,6-diphenyl-3-cyanopyridin-2-ylsulfanyl) acetamide (VIk).** Mass spectrum, *m*/*z* (I_{rel}, %): 465 (2) $[M + 2]^+$, 464 (4) $[M + 1]^+$, 463 (12) $[M]^+$, 462 (6) $[M - 1]^+$, 329 (35) $[M - AcC_6H_4NH]^+$, 301 (100) $[M - PhNH]^+$, 227 (13), 120 (14), 91 (7) $[PhN]^+$, 77 (16) $[Ph]^+$, 43 (15) $[Ac]^+$. *M* 436.562.

Octyl-2-(4,6-diphenyl-3-cyanopyridin-2-ylsulfanyl) acetate (VIl). Mass spectrum, *m*/*z* (I_{rel}, %): 460 (4) $[M + 2]^+$, 459 (19) $[M + 1]^+$, 458 (57) $[M]^+$, 457 (90) $[M - 1]^+$, 301 (100) $[M - Me(CH_2)_7OCO]^+$, 288 (11), 255 (15), 227 (13), 140 (6), 77 (16) $[Ph]^+$, 69 (9), 55 (18), 43 (47), 41 (39). *M* 458.627.

Nonyl-2-(4,6-diphenyl-3-cyanopyridin-2-ylsulfanyl) acetate (VIm). Mass spectrum, *m*/*z* (I_{rel}, %): 474 (4) $[M + 2]^+$, 473 (16) $[M + 1]^+$, 472 (48) $[M]^+$, 471 (44) $[M - 1]^+$, 301 (100) $[M - Me(CH_2)_8OCO]^+$, 288 (9), 255 (12), 227 (11), 77 (8) $[Ph]^+$, 55 (17), 43 (39). *M* 472.655.

3-Amino-2-acyl-4,6-diphenylthieno[2,3-*b*]pyridines (VIIa–VIIc) *(general procedure).* A mixture of 10 mmol of the corresponding 2-alkylsulfanylnicotinonitrile (**VIa–VIc**), 5.6 mL of 10 wt % aqueous KOH solution (10 mmol of the alkali), and 20 mL of DMF was stirred during 2 h and then diluted with equal volume of water. The formed precipitate was filtered off and washed with water, ethanol, and hexane.

3-Amino-2-(1-oxoisoamyl)-4,6-diphenylthieno-[2,3-*b*]pyridine (VIIa). Mass spectrum, *m*/*z* (I_{rel}, %): 387 (100) $[M + 1]^+$. *M* 386.519.

Isopropyl-2-(3-amino-4,6-diphenylthieno[2,3-*b*]-pyridin-2-yl) acetate (VIIb). Mass spectrum, *m*/*z* (I_{rel}, %): 389 (100) $[M + 1]^+$. ^{13}C NMR spectrum, δ_C, ppm: 22.28, 68.24, 95.98, 118.68, 120.84, 127.71, 129.11, 129.37, 129.78, 130.45, 136.82, 137.79, 147.76, 148.49, 156.80, 161.41, 164.45. *M* 388.491.

3-Amino-2-(1-oxoamyl)-4,6-diphenylthieno[2,3-*b*]-pyridine (VIIc). Mass spectrum, *m*/*z* (I_{rel}, %): 387 (100) $[M + 1]^+$. *M* 386.519. Fluorescent under UV irradiation.

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