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AZIRIDINYL KETONES AND THEIR CYCLIC ANILS.

9.* SUBSTITUTED 1,3-DIAZABICYCLO[3.1.0]HEX-3-ENES

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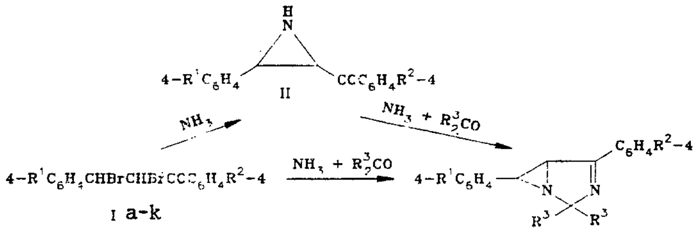
Aryl-substituted 1,3-diazabicyclo[3.1.0]hex-3-enes were obtained by the reaction of α,β-dibromodihydrochalcones with ammonia and formaldehyde (or acetone). Their structures were confirmed by spectral methods and x-ray diffraction analysis.

The interest in substituted 1,3-diazabicyclo[3.1.0]hex-3-enes is due to their photochromic properties [2-5]. It has been proposed that compounds with a p-nitrophenyl group in the 6 position of the two-ring system, for which the most profound changes in color are characteristic, be used to obtain light-sensitive materials, including materials for recording photographic and holographic images [6-11]. In the present research we set out to study the structure and the effect of various substituents on the properties of 1,3-diazabicyclo[3.1.0]hex-3-ene.

The method for the synthesis of the desired heterocycle proposed in [2, 6] was based on the reaction of aldehydes (or ketones) and ammonia with aziridinyl ketones II, which, in turn, are obtained from α,β-dibromodihydrochalcones I. The process can be accomplished in one step without isolation of aziridinyl ketones II; to obtain high yields ammonia should be passed into a suspension of dibromide I in methanol until the dibromide has dissolved completely, and the aldehyde or acetone should be added to the reaction system only after this while continuing saturation of the solution with ammonia:



IIIa-e,g; IVa,b,e-k

I, III, IV **a,c** $R^1 = H$, **b,d** $R^1 = Br$, **e – k** $R^1 = NO_2$; **a,b,e** $R^2 = H$, **f** $R^2 = Cl$, **c,d,g** $R^2 = Br$, **h** $R^2 = NO_2$, **i** $R^2 = CH_3$, **j** $R^2 = OCH_3$, **k** $R^2 = C_6H_5$; III $R^3 = H$, IV $R^3 = CH_3$

It was noted that the addition of formaldehyde (or acetone) at the start of the reaction leads to a decrease in the yield and contamination of the desired III and IV with

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*See [1] for Communication 8.

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TABLE 1. Characteristics of III and IV

Compound	Empirical formula	Color of the photocolored form	mp, °C)	UV spectrum, λ_{max}, nm ($\varepsilon \cdot 10^{-3}$)	IR spectrum (KBR), cm^{-1}				PMR spectrum,* δ, ppm (J, Hz)					Yield, %
					ν_{C-N}	$\nu^{as}_{NO_2}$	$\nu^{s}_{NO_2}$	$\nu^{as}_{N azir ring}$	CH_3	CH		CH_2	$J_{5,6}$	
										5-H	6-H			
IIa	$C_{16}H_{14}N_2$	Pink	127...128	245 (17,9)	1600	—	–	849	—	3,57	2,42	5,18	1,9	64
IIb	$C_{16}H_{13}BrN_2$	Pink	117...118	243 (25,0)	1610	—	—	854	—	3,52	—	5,17	—	77
IIIc	$C_{16}H_{13}BrN_2$	Pink	118...119	258 (22,9)	1606	—	—	862	—	3,47	2,36	5,13	1,95	47
IIId	$C_{16}H_{12}Br_2N_2$	Pink-red	121...122	256 (25,7)	1608	—	—	852	—	3,51	—	5,21	—	52
IIIe	—	Blue	179...180 (179...181 [2])	251 (17,0); 286 (13,0)	1609	1515	1345	867	—	3,60	2,51	5,24	1,7	97
IIIg	$C_{16}H_{12}Br_2N_3O_2$	Green	162...163	267 (27,0)	1595	1512	1342	857						85
IVa	$C_{18}H_{18}N_2$	—	84	246 (20,0)	1604	—	—	842	1,53	3,51	2,49	—	1,7 -	15
IVb	$C_{18}H_{17}BrN_2$	—	128...129	242 (24,0)	1603	—	—	849						
IVe	—	Dark blue	187 (187...188 [7], 182...183 [2])	252 (19,0); 283 (13,0)**	1602	1520	1350	860	1,61; 1,60	3,62	2,62	—	1,7***	42 77
IVf	$C_{18}H_{16}ClN_3O_2$	Dark blue	170	258 (19,0)	1602	1518	1344	862	1,52	3,64	2,69	—	~2	70
IVg	$C_{18}H_{16}BrN_3O_2$	Dark blue	172	261 (28,0)	1597	1512	1349	860	1,53	3,53	2,56	—	~2	60
IVh	$C_{18}H_{16}N_4O_4$	Dark blue	165	275 (41,0)	1602	1516	1345	862						48
IVi	$C_{19}H_{19}N_3O_2$	Dark blue	177...178	263 (22,3)	1600	1510	1342	867						71
IVj	$C_{19}H_{19}N_3O_3$	Dark blue	184	279 (27,5)	1609	1542	1345	858						68
IVk	$C_{24}H_{21}N_3O_2$	Dark blue	180...182	291 (40,5)	1598	1511	1344	862	1,46	3,56	2,59	—	~2	35

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*$J_{2,5}$ = 2.2 Hz for IIIa, b, d, e.

**According to the data in [6], λ^{EtOH}, nm ($\varepsilon \cdot 10^{-3}$): 250 (30.0), 283 (23.0); according to the data in [21]. $\lambda^{CH_2Cl_2}$, nm ($\varepsilon \cdot 10^{-3}$): 250 (14.0), 289 (12.05).

***According to the data in [2], δ 5-H 3.63, 6-H 2.63 ppm, $J_{5,6}$ 1.8 Hz.

TABLE 2. Coordinates of the Nonhydrogen Atoms in Structure IVe*

Atom	X	Y	Z	Atom	X	Y	Z
$N_{(1)}$	0,2336(2)	0,2553(2)	0,0928(2)	$C_{(13)}$	−0,4047(4)	0,2651(4)	−0,3225(4)
$C_{(2)}$	0,2432(3)	0,1407(3)	−0,0361(3)	$C_{(14)}$	−0,2556(3)	0,2654(3)	−0,2113(4)
$N_{(3)}$	0,0894(2)	0,1035(2)	−0,1710(2)	$C_{(15)}$	0,1480(3)	0,3014(3)	0,3143(3)
$C_{(4)}$	0,0016(3)	0,1888(3)	−0,1426(3)	$C_{(16)}$	0,0744(3)	0,2510(3)	0,3937(3)
$C_{(5)}$	0,0765(3)	0,2894(3)	0,0194(3)	$C_{(17)}$	0,0919(3)	0,3266(3)	0,5428(3)
$C_{(6)}$	0,1196(3)	0,2200(3)	0,1505(3)	$C_{(18)}$	0,1838(3)	0,4529(3)	0,6102(3)
$C_{(7)}$	0,2977(4)	0,0161(3)	0,0222(4)	$C_{(19)}$	0,2608(3)	0,5051(3)	0,5361(3)
$C_{(8)}$	0,3537(3)	0,2023(3)	−0,0906(4)	$C_{(20)}$	0,2423(3)	0,4279(3)	0,3868(3)
$C_{(9)}$	−0,1560(3)	0,1889(3)	−0,2580(3)	$N_{(21)}$	0,1967(3)	0,5357(3)	0,7661(3)
$C_{(10)}$	−0,2064(3)	0,1134(3)	−0,4165(3)	$O_{(22)}$	0,2702(3)	0,6517(3)	0,8200(3)
$C_{(11)}$	−0,3537(4)	0,1156(4)	−0,5252(4)	$O_{(23)}$	0,1284(3)	0,4872(2)	0,8306(3)
$C_{(12)}$	−0,4527(4)	0,1911(4)	−0,4785(5)				

*The coordinates of the hydrogen atoms and the temperature factors can be obtained from the authors.

impurities. It might be assumed that these carbonyl components hinder the formation of the aziridinyl ketones, which are required intermediates in the process. In [2] it was proposed that aziridinyl ketones II subsequently primarily react with the aldehydes (ketone) to give the azacetal form at the aziridine ring. However, our studies showed that, in the absence of ammonia, the aziridinyl ketones do not react with the aldehydes, probably because of strong intramolecular H bonding of the proton of the aziridine ring [12]. In our opinion, a more likely primary process is attack by a molecule of ammonia on the keto group of II with subsequent reaction of the imino group of the heteroring with the aldehyde (ketone) and subsequent cyclization to imidazolines III (or IV).

A number of characteristic bands, viz., $\nu_{C=N}$, $\nu_{NO_2}{}^{s}$, and $\nu_{NO_2}{}^{as}$ and ν^{s} and ν^{as} of the aziridine ring, are observed in the IR spectra of III and IV. In the spectra of model 2,2-dimethyl-4-aryl-Δ^3-imidazoline [13] and -Δ^3-oxazoline [14] derivatives of the $\nu_{C=N}$ values lie at 1630-1667 cm^{-1}. When an aziridine ring is introduced, this frequency decreases by 30-50 cm^{-1},* as a consequence of which the $\nu_{C=N}$ values for III and IV lie in the region of the absorption of aromatic fragments, and it is not possible to separate the $\nu_{C=C}$ and $\nu_{C=N}$ bands. In addition, $\nu_{azirid}{}^{s}$ bands at 1210-1223 cm^{-1} develop in the spectra; in the case of III this band has lower intensity as compared with the same band in the spectra of IV. A band that can be correlated with the vibrations of an imidazoline fragment [5] is observed at 1048-1065 cm^{-1}. A characteristic δ^{s} doublet of a gem-dimethyl group at 1385 ± 6 and 1370 ± 4 cm^{-1} is also observed for IV.

The electronic spectra of III and IV are sums of the spectra of the isolated chromophoric fragments – aryl and arylazomethine – as evidenced by comparison of them with the spectra of the model compounds $R^1C_6H_4CH_3$ and $R^2C_6H_4C{=}NCH_3$ [15].

In the PMR spectra (100 MHz) of III the protons of the CH_2 group and the 5-H proton form an ABX system with virtual coupling. In the AB part (at 100 MHz) one observes a degenerate "doublet" at δ 5.17-5.28 ppm; this is possible for the small values $\Delta\nu_{AB}$ = 2-3 Hz, J_{BX} = 2 Hz, and J_{AX} = 0 Hz [16, p. 186]. This does not make it possible to obtain accurate parameters from a direct analysis of the ABX system. In recording the spectra of IIIa, c at 270 MHz, when $\Delta\nu_{AB}$ = 18 Hz, splitting due to coupling of this proton with one of the protons of the methylene group is displayed in the spectrum of the 5-H proton, and analysis of this ABX system by means of the rules of first-order spectra becomes possible; this analysis makes it possible to obtain J_{AB} = 16.6 Hz and J_{BX} = 2.2 Hz. This character of the spectrum confirms the presence in III molecules of a Δ^3-imidazoline ring that is stabilized (as compared with the alternative Δ^2-imidazoline structure) due to conjugation in the $N_{(3)}{=}C{-}C_6H_4{-}R^2$ fragment. For all III and IV the spectrum of the 6-H proton due to

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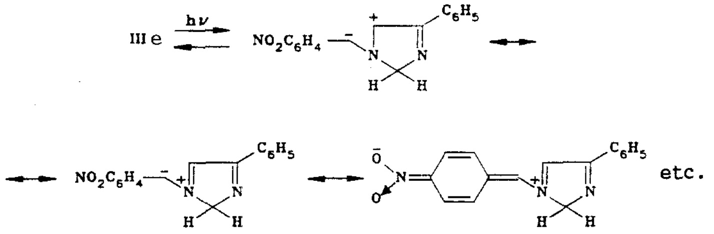
*For comparison, $\nu_{C=N}$ = 1609 cm^{-1} in the spectrum of IVj and 1630 cm^{-1} in the spectrum of 5-methoxy-4-(4-methoxyphenyl)-2,2-dimethyl-5-trifluoromethyl-Δ^3-1,3-oxazoline [14]. In their discussion of the Raman spectrum of IVe Padwa and coworkers [13] present a $\nu_{C=N}$ value of 1669 cm^{-1}; however, a weak band that probably has an overtone nature is observed in the IR spectra of only individual III and IV at 1660-1680 cm^{-1}.

TABLE 3. Bond (β) and Torsion (τ) Angles of the Bicyclic Part of the IVe Molecule

Angle	β°	Angle	β°	Angle	τ°
$C_{(5)}C_{(4)}N_{(3)}$	113,2(2)	$N_{(1)}C_{(6)}C_{(15)}$	115,7(2)	$N_{(1)}C_{(2)}N_{(3)}C_{(4)}$	6,5(3)
$C_{(9)}C_{(4)}N_{(3)}$	123,3(2)	$C_{(5)}N_{(1)}C_{(6)}$	61,3(2)	$C_{(2)}N_{(3)}C_{(4)}C_{(5)}$	−4,6(3)
$C_{(9)}C_{(4)}C_{(5)}$	123,4(2)	$C_{(5)}N_{(1)}C_{(2)}$	104,1(2)	$N_{(3)}C_{(4)}C_{(5)}N_{(1)}$	1,0(3)
$C_{(4)}N_{(3)}C_{(2)}$	108,9(2)	$C_{(2)}N_{(1)}C_{(6)}$	114,3(2)	$C_{(4)}C_{(5)}N_{(1)}C_{(2)}$	3,0
$C_{(4)}C_{(5)}N_{(1)}$	105,5(2)	$N_{(1)}C_{(2)}C_{(7)}$	112,1(2)	$C_{(5)}N_{(1)}C_{(2)}N_{(3)}$	−5,6(2)
$C_{(4)}C_{(5)}C_{(6)}$	112,5(2)	$C_{(7)}C_{(2)}C_{(8)}$	112,7(2)	$N_{(1)}C_{(6)}C_{(15)}C_{(20)}$	19,9
$C_{(6)}C_{(5)}N_{(1)}$	59,2(2)	$C_{(8)}C_{(2)}N_{(3)}$	107,9(2)	$C_{(4)}C_{(5)}C_{(6)}C_{(15)}$	−160,7(3)
$C_{(5)}C_{(6)}C_{(15)}$	119,6(2)	$N_{(3)}C_{(2)}N_{(1)}$	107,8(2)	$N_{(3)}C_{(4)}C_{(9)}C_{(10)}$	−13,3(4)
$C_{(5)}C_{(6)}N_{(1)}$	59,5(2)				

its coupling with the 5-H proton shows up in the form of a doublet with spin-spin coupling constant (SSCC) 1.70-1.95 Hz; this reflects the trans orientation of the aziridine protons.

The development of a color is observed when crystals of III and IV are irradiated with UV light (see Table 1). The photochromism is due to cleavage of the C—C bond of the aziridine ring with the formation of 1,3-dipolar ylid forms (azomethine ylids):



Compounds IIIe, g and IVe-k (R^1 = NO_2) are characterized by the greatest photosensitivity and the most profound changes. A low rate of dark conversion to the starting molecular form is also characteristic for them. These data reflect the fundamental role of the nitro group in stabilization of the ylid.

The time of the dark process for IVe is ~24 h at 20°C and 3-5 min at 100°C. It decreases appreciably when electron-acceptor substituents R^2 are introduced; for example, for IVh this time is 1.5 h at 20°C. Substituents R^2 also have a slight effect on the tint of the color of the ylid form (from green to intense dark blue). In crystals of compounds that contain R^1 = NO_2 measurement of the maximum possible number of photocycles is hindered because of the retarded character of the dark process, but prolonged observations showed that visible signs of a change in the composition of the compounds was not observed even after 100 photocycles.

The stabilities of the ylids of diazabicyclohexenes III and IV decrease sharply on passing from the solid state to solutions as a consequence of their rapid isomerization to dihydropyrazines [17]. The fact that composites, upon irradiation of which the dark-blue color of the ylid, which is converted irreversibly to a yellow color, develops, are obtained when III and IV are introduced in dissolved form into a colorless polymeric matrix [poly(methyl) methacrylate] is also explained by secondary processes. The introduction into the polymer of the same compounds in dispersed form makes it possible to obtain materials that retain photoreversibility.

It seemed of interest to make a more detailed study of the structures of the investigated molecules. For this, we realized the x-ray diffraction analysis of IVe* (see Tables 2 and 3 and Fig. 1). The bond angles and bond lengths in IVe are close to the values obtained for compounds with similar structures: 2-(4-bromophenyl)-1,3-diazabicyclo[3.1.0]-hexene (V) [19] and 2-carbamoylmethyl-1,3-diazabicyclo[3.1.0]hexan-4-one (VI) [20]. The

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*The investigation was carried out at A. Mickiewicz University (Poznan, Poland) within the framework of an interuniversity collaboration between this university and Kharkov State University.

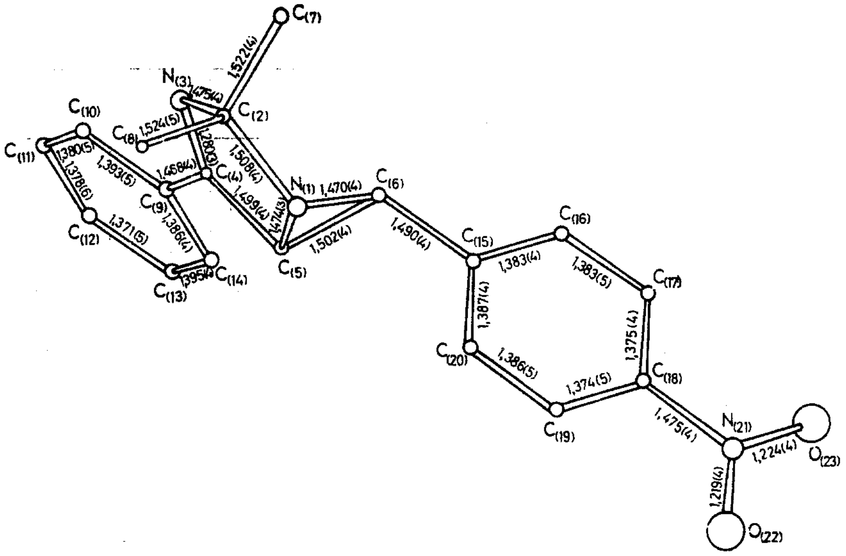

Fig. 1. Structure of the 2,2-dimethyl-6-(4-nitrophenyl)-4-phenyl-1,3-diazabicyclo[3.1.0]hex-3-ene (IVe) molecule.

$C_{(5)}$–$C_{(6)}$ bond in the aziridine ring is somewhat longer than the bond in both aziridine (1.481 Å [18]) and V (1.460 Å) and VI (1.485 Å).

The imidazoline ring in the IVe molecule has an envelope conformation with deviation of the $C_{(2)}$ atom from the $N_{(3)}C_{(4)}C_{(5)}N_{(1)}$ plane in a direction opposite from the $C_{(6)}$ atom of the aziridine ring, as follows from an analysis of the torsion angles of the five-membered ring. The aziridine ring forms an angle of 46.8° with the mean plane of the imidazoline ring. The plane of the benzene ring in the phenylimidazoline fragment is turned by 12.6°. The dihedral angle between the nitrophenyl fragment and the aziridine ring is 75.4°. The high degree of coplanarity of the phenylimidazoline part of the molecule is due to the favorable combination of electronic and steric factors. In turn, the observed orientation of the nitrophenyl ring with respect to the aziridine ring constitutes evidence for the manifestation of interaction of the aromatic π system with the "banan" σ bonds of the three-membered ring, which is confirmed by the shortening of the $C_{(6)}$–$C_{(15)}$ bond to 1.490 Å.

The x-ray diffraction data (Fig. 1) confirm a trans orientation of the nitrophenyl radical with respect to the imidazoline ring. It also follows from these data that the bond lengths and bond angles in the five-membered ring are close to the normal values, i.e., this ring should not change substantially upon photoelectronic opening of the aziridine ring.

EXPERIMENTAL

The IR spectra of KBr pellets of the compounds were obtained with a Specord IR-75 spectrometer. The electronic absorption spectra of solutions in methanol with substance concentrations of $(2\text{-}4)\cdot 10^{-5}$ mole/liter were obtained with a Specord UV-vis spectrophotometer. The PMR spectra of solutions in $CDCl_3$ were recorded with Tesla BS-567A (60 MHz) and Bruker WP-270 (270 MHz) spectrometers with tetramethylsilane (TMS) as the internal standard. The course of the reactions and the purity of the compounds obtained were monitored by TLC on Silufol UV-254 plates in chloroform–ethyl acetate (3:2). The results of elementary analysis of III and IV for N were in agreement with the calculated values.

X-Ray Diffraction Analysis of IVe. The crystals had triclinic syngony with the following unit cell parameters: $a = 9.551(1)$, $b = 10.109(1)$, $c = 9.538(1)$ Å, $\alpha = 103.92(1)°$, $\beta = 113.52(1)°$, $\gamma = 91.96(1)°$, $v = 810.8(2)$ Å³, $d_{calc} = 1.26$ g/cm³, $Z = 2$, $\mu_{CuK\alpha} = 5.99$ cm^{-1}, space group P$\bar{1}$. The measurement of the intensities of the beams of the diffraction pattern of this compound was carried out at 20°C with a Syntex P2_1 four-circle automatic diffractometer (CuK_α emission, graphite monochromator) using the method of $\theta/2\theta$ scanning up to $2\theta_{max} = 116°$. We obtained 1929 independent reflections with $I > 2\sigma_I$. The structure was decoded by the direct method by means of the MULTAN-80 program and was refined by the method of least squares within the total matrix anisotropic approximation by means of the

SHELX-76 program. All of the hydrogen atoms were revealed in differential synthesis. The final divergence factor R = 5.05 (R_W = 5.05) with respect to 1929 reflections with $F^2 \geq 3\sigma$.

4-Phenyl-6-(4-bromophenyl)-1,3-diazabicyclo[3.1.0]hex-3-ene (IIIb). Dry ammonia was passed through a suspension of 8.94 g (0.02 mole) of 1-phenyl-3-(4-bromophenyl)-2,3-dibromo-1-propanone (Ib) in 50 ml of methanol until α,β-dibromodihydrochalcone Ib (R_f 0.73) had vanished in the reaction mixture, after which 1.80 g (0.02 mole) of paraformaldehyde was added in portions while the passage of ammonia into the mixture was continued. The passage of ammonia was discontinued after the paraformaldehyde had dissolved completely, and the flask was sealed and allowed to stand at room temperature in the dark for 5 days. The resulting precipitate was removed by filtration, and washed successively with 200 ml of water and 10 ml of methanol, and crystallized from methanol—acetone (1:1) to give 4.82 g (77%) of compound IIIb (R_f 0.35), mp 117-118°C.

Compounds IIIa,c-e,g. These substances were obtained similarly.

2,2-Dimethyl-6-(4-nitrophenyl)-4-(4-methoxyphenyl)-1,3-diazabicyclo[3.1.0]hex-3-ene (IVj). Dry ammonia was passed through a suspension of 4.43g (0.01 mole) of 3-(4-nitrophenyl)-1-(4-methoxyphenyl)-2,3-dibromo-1-propanone (Ij) in 30 ml methanol until α,β-dibromodihydrochalcone Ij (R_f 0.67) had vanished in the reaction mixture. Then 5 ml (3.95 h, 68 mmole) acetone was added, and more ammonia passed through for 40 min. The reaction mixture turned a deep orange color. The flask was sealed and left at room temperature overnight, then left for 2-3-days in a condensor.

Compounds IVb, e-k. These substances were similarly obtained.

For the isolation of IVa the solvent was removed by distillation to dryness with a rotary evaporator, and the product was extracted from the residue with warm hexane. The extract was passed through a column (2 by 60 cm) packed with silica gel 100/250 by elution with hexane, and the solvent was evaporated until crystals developed. Cooling gave IVa with mp 84°C.

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