**Table I.** Effect of 6-Selenoguanosine, α-2′-Deoxy-6-thioguanosine, β-2′-Deoxy-6-thioguanosine, α-2′-Deoxy-6-selenoguanosine, and β-2′-Deoxy-6-selenoguanosine on the Growth of L-5178Y

| | % survival | | |
|---|---|---|---|
| Control 100% | $1.0 \times 10^{-4}$ *M* | $1.0 \times 10^{-5}$ *M* | $1.0 \times 10^{-6}$ *M* |
| 6-Selenoguanosine | 4 | 8 | 35 |
| α-2′-Deoxy-6-thio-guanosine | 18 | 65 | 73 |
| β-2′-Deoxy-6-thio-guanosine | 10 | 13 | 34 |
| α-2′-Deoxy-6-seleno-guanosine | 66 | 78 | 88 |
| β-2′-Deoxy-6-seleno-guanosine | 12 | 16 | 50 |

## Experimental Section‡

**2-Amino-6-seleno-9-(2′-deoxy-3′,5′-di-*O*-*p*-toluoyl-β-D-*erythro*-pentofuranosyl)-9*H*-purine (2).** Condensed $H_2Se$§ (1.62 ml) was bubbled through a soln of 0.80 g (0.0035 g-atom) of Na in 300 ml of abs MeOH. 2-Acetamido-6-chloro-9-(2′-deoxy-3′,5′-di-*O*-*p*-toluoyl-β-D-*erythro*-pentofuranosyl)-9*H*-purine **(1)** (2.26 g, 0.004 mole) was introduced into the well-stirred soln. The mixture was stirred under $N_2$ at room temp for 3 days. The greenish solid was collected by filtration and washed with MeOH (10 ml). The residue (2.49 g) was recrystd from MeOH to give 1.53 g (75%) of the product: mp 133–137°; uv $\lambda_{max}^{MeOH}$ 357.5 ($\epsilon_{max}$ 11,940), 239 nm (40,660); $[\alpha]^{25}D$ −88.4° (*c* 0.206, MeOH). The analytical sample was recrystd from MeOH. *Anal.* ($C_{26}H_{25}N_5SeO_5 \cdot H_2O$) C, H, N. The elemental analysis suggested that compound **2** is a hygroscopic hydrate.

**2-Amino-9-(2′-deoxy-β-D-*erythro*-pentofuranosyl)-9*H*-purine-6-selenol (β-2′-Deoxy-6-selenoguanosine) (3).** Partially protected β-2′-deoxy-6-selenoguanosine **(2)** (1.65 g, 0.003 mole) was introduced into a soln of 0.207 g of Na (0.009 g-atom) in 50 ml of abs MeOH, and the mixture was stirred and kept overnight under $N_2$. The reaction mixture was concentrated to dryness under reduced pressure. The residue was dissolved in 50 ml of ice-cold $H_2O$ and the soln was extracted with $CHCl_3$ (5 × 40 ml). The aqueous layer was clarified by filtration. The clear yellow filtrate was acidified (pH 4–5) with AcOH and kept 30 min in an ice bath. The yellow solid was filtered off, washed with 5 ml of cold $H_2O$ and 10 ml of $Et_2O$, and dried to give 0.57 g (54%) of **3**: mp 166–167° (bubbling). Reprecipitation of **3** from $Na_2CO_3$ soln did not purify further the product because of its instability in aqueous soln. On tlc# the $R_f$ value in $H_2O$ is 0.42: uv $\lambda_{max}^{pH\ 1.0}$ 370.5 ($\epsilon_{max}$ 21,100), 270 nm (6100); $\lambda_{max}^{H_2O}$ 358 ($\epsilon_{max}$ 25,800), 263.5 nm (6200); $\lambda_{max}^{pH\ 11.0}$ 330 ($\epsilon_{max}$ 18,100), 225 nm (11,950). *Anal.* ($C_{10}H_{13}O_3N_5Se \cdot H_2O$) C, H, N.

**2-Acetamido-6-seleno-9-(2′-deoxy-3′,5′-di-*O*-*p*-toluoyl-α-D-*erythro*-pentofuranosyl)-9*H*-purine (5).** Condensed $H_2Se$ (1.0 ml) was bubbled through a soln of 0.3 g (0.013 g-atom) of Na in 60 ml of abs EtOH. 2-Acetamido-6-chloro-9-(2′-deoxy-3′,5′-di-*O*-*p*-toluoyl-α-D-*erythro*-pentofuranosyl)-9*H*-purine **(4)** (2.2 g, 0.0039 mole) in 40 ml of abs EtOH was introduced into the well-stirred soln. The mixture was stirred under $N_2$ at room temp for 80 min. The greenish solid was collected by filtration and washed with EtOH (10 ml). The residue was recrystd from 100 ml of EtOH to give 1.6 g (67.4%) of **5**: mp 139°; uv $\lambda_{max}^{MeOH}$ 361.5 ($\epsilon_{max}$ 16,340), 239 nm (45,320); $[\alpha]^{25}D$ −17.16° (*c* 0.204, MeOH). *Anal.* ($C_{28}H_{27}N_5SeO_6$) C, H, N.

**2-Amino-9-(2′-deoxy-α-D-*erythro*-pentofuranosyl)-9*H*-purine-6-selenol (α-2′-Deoxy-6-selenoguanosine) (6).** 2-Acetamido-6-seleno-9-(2′-deoxy-3′,5′-di-*O*-*p*-toluoyl-α-D-*erythro*-pentofuranosyl)-9*H*-purine **(5)** (1.5 g, 0.0025 mole) was introduced into a soln of Na (0.13 g, 0.0057 g-atom) in 70 ml of abs MeOH, and the mixture was stirred and kept overnight under $N_2$. The reaction mixture was concentrated to dryness under reduced pressure. The residue was dissolved in 15 ml of ice-cold $H_2O$, and the soln was extracted with $CHCl_3$ (5 × 20 ml). The aqueous layer was clarified by filtration. After the clear, yellow soln was acidified (pH 5–6) with AcOH and kept 1 hr at 0°, the yellow solid was filtered off, washed with 2–3 ml of cold $H_2O$ and 10 ml of $Et_2O$, and dried to give 0.6 g (70%) of **6**: mp 176° (bubbling). Because of the high solubility of the compound in $H_2O$, it is important to use a minimum amount of ice-cold $H_2O$ for the acid precipitation. On tlc# the $R_f$ value in $H_2O$ was 0.42: uv $\lambda_{max}^{pH\ 1.0}$ 371 ($\epsilon_{max}$ 21,900), 270 nm (5700); $\lambda_{max}^{H_2O}$ 357 ($\epsilon_{max}$ 25,210), 262.5 nm (5810); $\lambda_{max}^{pH\ 11.0}$ 330 ($\epsilon_{max}$ 18,170) 254 nm (11,460). *Anal.* ($C_{10}H_{13}O_3N_5Se \cdot H_2O$) C, H, N.

**Effects on Cultured Mouse Leukemia Cells.** The preliminary results of the tissue culture studies using L-5178Y cells are shown in Table I. The cell viability was determined by the dilute agar colony method.[6] 6-Selenoguanosine, α-2′-deoxy-6-thioguanosine, β-2′-deoxy-6-thioguanosine, α-2′-deoxy-6-selenoguanosine **(6)**, and β-2′-deoxy-6-selenoguanosine **(3)** inhibited cell division and caused cell death over a range from $1.0 \times 10^{-4}$ to $1.0 \times 10^{-6}$ mole after 2-hr incubation. β-2′-Deoxy-6-selenoguanosine **(3)** was found to have activity approximately equal to β-2′-deoxy-6-thioguanosine, but the α-seleno derivative **6** was much less active than α-2 -deoxy-6-thioguanosine. Further study of these compounds is in progress. Because of the instability of 2′-deoxy-6-selenoguanosine, fresh solutions of these compounds were prepared for each use in biological studies.

**Acknowledgments.** The authors are indebted to Dr. M. Y. Wang Chu for carrying out the tissue culture study, and to Drs. R. E. Parks, Jr., and P. Calabresi for their encouragement during the course of this investigation.

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‡All melting points are uncorrected. Analyses were carried out at Micro-Analysis, Inc., Marshallton, Wilmington, Del., and MidWest Microlab, Inc., Indianapolis, Ind.

§98.0% minimum purity $H_2Se$ from the Matheson Co., Inc., East Rutherford, N. J. 07073.

#Polygram CEL 300 PEI from Brinkmann Instruments, Inc., Westbury, N. Y.

# *New Compounds*

## Terpene Compounds as Drugs. 13.[1] *o*-Terpenylaminomethylphenols and Their *N*-Methyl Derivatives

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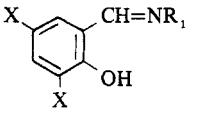
*Received February 25, 1972*

The interesting properties of several phenol derivatives and terpenoid compds used in the therapy of respiratory tract diseases have been recognized for a long time.[2] In a search for novel expectorant and antitussive agents, we synthesized a series of *o*-terpenylaminomethylphenols and their *N*-methyl derivatives (II, X = H) (Table II). Besides, in view of some similarity between these structures and the expectorant bromhexine[3] (*N*-cyclohexyl-*N*-methyl-2-amino-3,5-dibromobenzylamine), we also prepared compds II, where X = Br or Cl. N-Substituted salicylidenimines (I) were obtained by condensing the appropriate salicylaldehyde with the terpenylamine. Compds I were reduced to secondary amines (II), a number of which were N-methylated with HCHO–HCOOH.

Table I. *N*-Terpenylsalicylidenimines

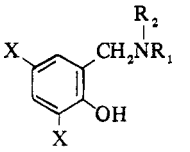


I

| No. | $R_1$ | X | Reflux time, hr | Yield, %[a] | Bp (mm) or mp, °C | Formula | Analyses |
|---|---|---|---|---|---|---|---|
| 1 | *trans*-2,2,6-Trimethylcyclohexyl | H | 3 | 85 | 118–120 (0.25) | $C_{16}H_{23}NO$ | C, H, N |
| 2 | *trans*-2,2,6-Trimethycyclohexyl | Cl | 3 | 83 | *b* | $C_{16}H_{21}Cl_2NO$ | C, H, Cl, N |
| 3 | *trans*-2,2,6-Trimethylcyclohexyl | Br | 3 | 86 | 125–126[c] | $C_{16}H_{21}Br_2NO$ | C, H, Br, N |
| 4 | α-Fenchyl | H | 3 | 79 | 120–122 (0.02)[d] | $C_{17}H_{23}NO$ | C, H, N |
| 5 | α-Fenchyl | Cl | 3 | 80 | *b* | $C_{17}H_{21}Cl_2NO$ | C, H, Cl, N |
| 6 | α-Fenchyl | Br | 4 | 85 | *b* | $C_{17}H_{21}Br_2NO$ | C, H, Br, N |
| 7 | Bornyl | H | 5 | 80 | 134–136 (0.04) | $C_{17}H_{23}NO$ | C, H, N |
| 8 | Bornyl | Cl | 5 | 85 | *b* | $C_{17}H_{21}Cl_2NO$ | C, H, Cl, N |
| 9 | Bornyl | Br | 5 | 92 | *b* | $C_{17}H_{21}Br_2NO$ | C, H, Br, N |
| 10 | Menthyl | H | 3 | 75 | 122–124 (0.03)[e] | $C_{17}H_{25}NO$ | C, H, N |
| 11 | Menthyl | Cl | 5 | 80 | *b* | $C_{17}H_{23}Cl_2NO$ | C, H, Cl, N |
| 12 | Menthyl | Br | 5 | 85 | *b* | $C_{17}H_{23}Br_2NO$ | C, H, Br, N |

[a]Crude product. [b]Thick oil. [c]Crystallized from petroleum ether (bp 40–70°). [d]See ref 10. [e]See ref 11.

Table II. *o*-Terpenylaminomethylphenols and Their *N*-Methyl Derivatives



II

| No. | $R_1$ | $R_2$ | X | Yield, % | Mp, °C[a] | $[\alpha]^{20}D$,[b] deg | Formula[c] |
|---|---|---|---|---|---|---|---|
| 13 | *trans*-2,2,6-Trimethylcyclohexyl | H | H | 65 | 200–201[d] | | $C_{16}H_{25}NO \cdot HCl$ |
| 14 | *trans*-2,2,6-Trimethylcyclohexyl | H | Cl | 81 | 203–205[e] | | $C_{16}H_{23}Cl_2NO \cdot HCl$ |
| 15 | *trans*-2,2,6-Trimethylcyclohexyl | H | Br | 65 | 213–215[e] | | $C_{16}H_{23}Br_2NO \cdot HCl$ |
| 16 | α-Fenchyl | H | H | 68 | 193–194[f] | +13 | $C_{17}H_{25}NO \cdot HCl$ |
| 17 | α-Fenchyl | H | Cl | 77 | 199–200[e] | +12.5 | $C_{17}H_{23}Cl_2NO \cdot HCl$ |
| 18 | α-Fenchyl | H | Br | 46 | 206–207[e] | +10 | $C_{17}H_{23}Br_2NO \cdot HCl$ |
| 19 | Bornyl | H | H | 45 | 178–179 | | $C_{17}H_{25}NO \cdot HCl$ |
| 20 | Bornyl | H | Cl | 57 | 214–215 | | $C_{17}H_{23}Cl_2NO \cdot HCl$ |
| 21 | Bornyl | H | Br | 72 | 220–221 | | $C_{17}H_{23}Br_2NO \cdot HCl$ |
| 22 | Menthyl | H | H | 66 | 202–203 | −68 | $C_{17}H_{27}NO \cdot HCl$ |
| 23 | Menthyl | H | Cl | 78 | 216–217[d] | −35 | $C_{17}H_{25}Cl_2NO \cdot HCl$ |
| 24 | Menthyl | H | Br | 57 | 212–214 | −34 | $C_{17}H_{25}Br_2NO \cdot HCl$ |
| 25 | Bornyl | $CH_3$ | H | 69[g] | 159–160 | | $C_{18}H_{27}NO \cdot HCl$ |
| 26 | Bornyl | $CH_3$ | Cl | 56[h] | 175–176 | | $C_{18}H_{25}Cl_2NO \cdot HCl$ |
| 27 | Bornyl | $CH_3$ | Br | 73[h] | 170–172 | | $C_{18}H_{25}Br_2NO \cdot HCl$ |
| 28 | Menthyl | $CH_3$ | H | 70[i] | 175–176 | −29 | $C_{18}H_{29}NO \cdot HCl$ |
| 29 | Menthyl | $CH_3$ | Cl | 86[i] | 174–175 | −34 | $C_{18}H_{27}Cl_2NO \cdot HCl$ |
| 30 | Menthyl | $CH_3$ | Br | 86[i] | 178–179 | −30 | $C_{18}H_{27}Br_2NO \cdot HCl$ |

[a]All compounds were isolated as HCl salts in $Et_2O$ soln using dry HCl. [b]EtOH (*c* 2). [c]All analyses were for C, H, N. [d]Recrystd from AcOEt-EtOH. [e]Recrystd from dioxane. [g]Reaction time 48 hr. [h]Reaction time 90 hr. [i]Reaction time 24 hr.

Antitussive tests[4] indicated that only compounds 27 and 28 were active, their potency being distinctly inferior to that of codeine. No significant expectorant activity[5] was observed for the title compds. In view of this, further evaluation in this area was not considered justified.

## Experimental Section†

**Chemistry. Intermediates.** Salicylaldehyde, 3,5-dibromosalicylaldehyde, and 3,5-dichlorosalicylaldehyde were commercial products. (±)-Bornylamine[6] and (−)-menthylamine[7] were prepd as previously described by Na–EtOH redn of (±)-camphor oxime and (−)-menthoxime, respectively. (−)-α-Fenchylamine was prepd by $H_2$–$PtO_2$ redn of (+)-fenchone oxime.[8]

†Bp are uncor. Mp are cor and were taken on a Büchi capillary mp apparatus. Satisfactory ir and nmr spectra were recorded for all new compds. The purity of the compds as well as the progress of the reactions was checked by tlc on silica gel $GF_{254}$ (E. Merck AG., Germany) using $C_6H_6$–MeOH (8:2), and detecting the spots by spraying with Dragendorff's reagent.

*trans*-**2,2,6-Trimethylcyclohexylamine.** A stirred soln of 3 moles of NaOH in 100 ml of $H_2O$ was cooled to −5°, and $Br_2$ (144 g, 0.9 mole) was added during a 40-min period. After 30 min stirring at 0°, *trans*-2,2,6-trimethylcyclohexanecarboxamide[9] (144 g, 0.85 mole) was added at 15–20°, and stirring was continued at room temp for 30 min. The mixt was then heated at 100° for 40 min, cooled, and extd with $Et_2O$. The $Et_2O$ soln was dried (NaOH) and concd, and the residue was distd to give 83 g (69% yield) of a colorless oil, bp 67–70° (14 mm). *Anal.* ($C_9H_{19}N$) C, H, N.

**General Methods. *N*-Terpenylsalicylidenimines (I) (Table I).** A soln of 0.1 mole of the appropriate salicylaldehyde and 0.1 mole of the terpenylamine in 100 ml of anhyd PhH was refluxed while the $H_2O$ azeotrope was removed with a takeoff adapter. After the $H_2O$ had been all removed, the organic soln was evapd to dryness, and the residue was distd or used directly in the next step.

***o*-Terpenylaminomethylphenols (II) (Table II).** A soln of 0.1 mole of the appropriate I in 300 ml of anhyd MeOH was stirred at room temp, while 0.2 mole of $NaBH_4$ was added portionwise. After 30 min stirring at room temp, the organic soln was poured into excess $H_2O$ and extd with $Et_2O$. The $Et_2O$ ext was washed ($H_2O$), dried ($MgSO_4$), and evapd to dryness, and the residue was used for further reaction without purification.

When final products, compds II were converted to their hydrochlorides and purified as shown in Table II.

*N*-Methyl Derivatives of *o*-Terpenylaminomethylphenols (Table II). To 0.1 mole of the appropriate II, 0.375 mole of 88% HCOOH, and 0.375 mole of a 35% HCHO soln were added with cooling. The mixt was first heated slowly and then refluxed for a time varying from 24 to 90 hr. It was then cooled and basified with 10% KOH soln, and the basic material was filtered off or extd with $Et_2O$ and worked up in the usual manner.

**Acknowledgment.** The authors wish to thank Dr. A. Gallazzi for analytical data and Mr. G. Bietti for his assistance in preparing the compounds.

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# Derivatives of 3,5-Dihydro-4*H*-benzo[2,3]diazepin-4-one†

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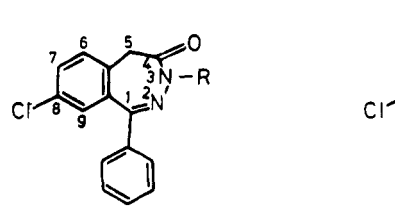
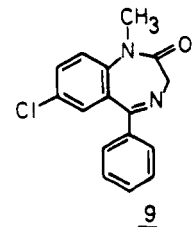
As part of a program for the synthesis and biological evaluation of potentially psychoactive compounds, we became interested in derivatives of 3,5-dihydro-4*H*-benzo[2,3]diazepin-4-one, in particular, in **1**, an isomer of diazepam.[1] Compounds having the title structure and bearing a hydrogen or methyl group, respectively, at C-1 have been made by condensation of 2-formyl- and 2-phenylacetic acids with hydrazine, but their biological properties have not been described.[2] We have prepared **1** and **2** by thermal cyclization of the methyl hydrazone and hydrazone, respectively, of 2-benzoyl-4-chlorophenylacetic acid. Likewise, compds **3** and **4** were prepared from 2-benzoyl-4,5-dimethoxyphenylacetic acid and compd **5** from 2-acetyl-4,5-dimethoxyphenylacetic acid. A water-soluble by-product was isolated in the last reaction and was assigned the 2-amino-3-isoquinolone structure **8**. The guanylhydrazone **11** of 2-acetyl-4,5-dimethoxyphenylacetic acid failed to cyclize to a 2,3-diazepine derivative. Treatment of **2** and **5** with an appropriate dialkylaminoalkyl halide afforded compds **6** and **7**, respectively.‡

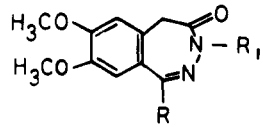
**Pharmacology.** The compds examined were suspended in a 0.2% agar suspension and given orally (po) or parenterally (ip) for evaluation of the neuropharmacological profile

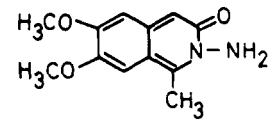




**9**

**1** R = $CH_3$
**2** R = H
**6** R = $CH_2CH_2CH_2N(CH_3)_2$





**8**

**3** R = Ph ; $R_1$ = $CH_3$
**4** R = Ph ; $R_1$ = H
**5** R = $CH_3$ ; $R_1$ = H
**7** R = $CH_3$ ; $R_1$ = $CH_2CH_2NEt_2$

in CF male mice.[4] Compd **8** showed dose-dependent CNS effects of sedation, ptosis, and ataxia, and its effective dose was 100 mg/kg po and 50 mg/kg ip. In contrast to compd **9**, muscular relaxation and hypotonia were not observed. Similar effects were observed with compd **5** at 250 mg/kg po and 100 mg/kg ip. Compds **1**, **3**, and **4** demonstrated sedation and ptosis at doses above 250 mg/kg po, but these effects were not dose dependent. Compd **1** at 50 mg/kg ip showed equal activity to compd **8**. However, compd **1** required 10 times the parenteral dose to produce equivalent CNS effects by the oral route. The lethal dose in mice for compds **1**, **3**, **4**, **5**, and **8** was more than 1000 mg/kg po.

Compd **2** showed no evidence of CNS activity up to 1000 mg/kg, whereas evidence of CNS stimulation was observed in compds **6** and **7**. The lethal dose of **6** and **7** was 1000 mg/kg po. In mice there was no evidence of antielectroshock activity, specific antagonism to mescaline-induced "scratch stereotypy" or antagonism to the acetic acid induced writhing phenomenon,[5] up to 100 mg/kg po.

In conclusion it appears that **1**, which differs from **9** in the disposition of $NCH_3$ and $CH_2$ groups, is biologically much less active. The diminished activity of **1** and other 2,3-benzodiazepine congeners described herein, compared to the 1,4-diazepine analogs may be due to decreased basicity of the former and/or different juxtaposition of potential binding sites. The disappointing results encountered for the 2,3-diazepine series discouraged us from expanding the project, although the present method would have easily permitted the synthesis of a larger number of analogs of **1** and **3**.

## Experimental Section§

**1-Phenyl-3-methyl-3,5-dihydro-8-chloro-4*H*-benzo[2,3]diazepin-4-one (1).** 2-Benzoyl-4-chlorophenylacetic acid# (2 g, 7.3 mmoles) and hydrazine hydrate (0.4 g, 8 mmoles) in EtOH (10 ml) were heated under reflux for 4 hr. Evaporation of EtOH gave the oily

†Contribution No. 286 from CIBA Research Centre, Bombay 63, India.

‡After this work was completed, Wermuth and Flammang[3] reported the synthesis of 1-phenyl derivatives of the title structure.

§Mps are uncorrected. All compds were analyzed for C, H, and N and gave results within ±0.4% of the theoretical values. Ir and nmr spectral data were consistent with the structures assigned.

#Obtained from 1-phenyl-1-hydroxy-6-chloroindan by $CrO_3$ oxidation according to Nizamuddin, *et al.*[6]