Samples were prepared for the spectrometer by dissolving them in a small amount of methanol (pyridine for the O_2 adduct), pipetting 1 drop of the solution onto an aluminum sample plate, and evaporating the solvent. The C 1s peak from the benacen ligand was used as an internal binding energy reference. It was assigned a binding energy of 285.0 eV. The Co $2p_{3/2}$ spectra were obtained by counting the photoelectrons for 40 sec/channel at 0.2-eV kinetic energy intervals. The reported binding energies are the positions of the peak maxima and the half-widths are the full widths at half-maximum (FWHM).

Results and Discussion

The cobalt(II) Schiff base complex Co(benacen) has a binding energy (BE) of 780.0 ± 0.1 eV and a FWHM of 4.0 eV. A shoulder appears on the high-BE side of this peak indicating the possible presence of a Co(III) impurity.[6] The Co(III) complex Co(benacen)pyNO_2 has a BE of 781.4 ± 0.1 eV and a FWHM of 3.0 eV. This BE is 1.4 eV higher in energy than that of the Co(II) species. This increase in BE with increasing formal oxidation state is similar to that observed by Kramer and Klein for iron.[7] The measured Co $2p_{3/2}$ BE for the oxygen adduct Co(benacen)pyO_2 is 781.4 ± 0.1 eV, which is equal to the value for the cobalt(III)-nitro complex. The half-width was also 3 eV. This result indicates that the Co^{III}-O_2^- formulation proposed by esr studies[2] is correct. Rodley and Robinson have reported the X-ray structure of this complex.[8] They found a Co-O-O bond angle of 126 (2)° and a O-O distance of 1.26 (4) Å. These values are also indicative of a superoxide ligand as the superoxide ion has a bond length of 1.28 Å.

The X-ray structure of Co(benacen)NO indicates that the Co-N-O bond is bent with an angle of 122.9 (8)°.[9] Bent metal-NO bonds are thought to be indicative of NO^-. An examination of the X-ray photoelectron spectra show the BE of the Co $2p_{3/2}$ electrons is 780.9 ± 0.1 eV (FWHM = 2.8 eV), close to the value found for the cobalt(III)-nitro complex and the O_2 adduct. It thus seems that the best formulation is similar to the oxygen complex or Co^{III}-NO^-.

The N 1s region of the spectrum of Co(benacen)NO was examined. Two peaks are observed with an intensity ratio of 1:2 corresponding to the NO^- ligand and the benacen ligand, respectively. The BE for the NO^- was found to be 400.1 eV. This is in good agreement with the N 1s BE of 400.7 eV reported by Finn and Jolly[10] for the NO^- ligand in the complex $[Co(NH_3)_5NO]Cl_2$.

Registry No. Co(benacen), 36466-12-3; Co(benacen)(py)NO_2, 36609-90-2; Co(benacen)(py)O_2, 36643-07-9; Co(benacen)NO, 25848-52-6.

Acknowledgment. This work was partially supported by ARPA through the Northwestern University Materials Research Center and by a National Science Foundation Grant(GP-28134) for the purchase of the electron spectrometer.

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(6) A figure of the Co $2p_{3/2}$ spectra is available from the authors on request.

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Magnetic Properties of Oxovanadium(IV) Complexes of *N*-(Hydroxyalkyl)salicylidinimines

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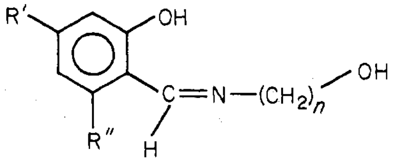
In recent years there has been considerable interest in the synthesis and magnetic properties of oxovanadium(IV) complexes with subnormal magnetic moments (referred to in this paper as subnormal complexes).[1-5] Recently Poddar, *et al.*,[6] have reported the synthesis of oxovanadium(IV) complex of *N*-(hydroxyethyl)salicylidinimine. They reported a room-temperature magnetic moment of 1.42 BM for this complex. Subsequently Kuge and Yamada[7] synthesized this compound independently and reported a room-temperature magnetic moment of 1.06 BM. Due to this anomaly in the literature it was of interest to repeat the work and study the magnetic properties of the complex in detail. In this note we report the magnetic properties of oxovanadium(IV) complexes of I.



I, R' = H, Cl, Br, NO_2, OCH_3
R'' = H, NO_2, OCH_3
n = 2, 3

Esr, infrared, and electronic spectral data of the complexes are also reported.

Experimental Section

Chemicals. Reagent grade salicylaldehyde and sodium acetate were obtained from J. T. Baker Chemical Co. Vanadyl dichloride was obtained from K & K Laboratories, Inc. 5-Chlorosalicylaldehyde, 5-bromosalicylaldehyde, 3-nitrosalicylaldehyde, 5-bromosalicylaldehyde, 3-nitrosalicylaldehyde, 5-nitrosalicylaldehyde, and propanolamine were purchased from Eastman Organic Chemicals. 3-Methoxysalicylaldehyde and 5-methoxysalicylaldehyde were obtained from Aldrich Chemical Co. Ethanolamine was the product of Matheson Coleman and Bell. All other chemicals used were of reagent grade quality.

Physical Measurements. Magnetic susceptibilities were determined by the Gouy method. Mercury tetrathiocyanatocobaltate was used as the standard. The diamagnetic corrections for the ligand and metal atoms were computed using a standard source.[8] The susceptibilities were corrected for TIP using a value of 50×10^{-6} cgsu. Electron spin resonance spectra were obtained with a Varian V-4502-12 X-band spectrometer using 100-kc modulation and a 9-in. electromagnet. Cylindrical quartz sample tubes and a Varian V-4533 cylindrical cavity were employed. For liquid nitrogen temperature a quartz dewar which fitted into the cavity was used. A minute sample of finely powdered diphenylpicrylhydrazil (DPPH) was used as a *g*-marker. Infrared spectra were obtained on a Perkin-Elmer Model 621 instru-

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Table I. Analytical Data of Oxovanadium(IV) Schiff Base Complexes

Complex	Color	Stoichiometry	% C		% H		% N	
			Calcd	Found	Calcd	Found	Calcd	Found
VO(Sal-ethanolamine)	Brown	$VC_9H_9NO_3$	46.98	47.62	3.94	4.39	6.09	5.77
VO(5-Cl-Sal-ethanolamine)	Brown	$VC_9H_8NO_3Cl$	40.86	41.24	3.05	3.06	5.30	5.07
VO(5-Br-Sal-ethanolamine)	Brown	$VC_9H_8NO_3Br$	34.98	35.20	2.61	2.89	4.53	4.24
VO(5-NO_2-Sal-ethanolamine)	Reddish brown	$VC_9H_8N_2O_5$	39.29	39.68	2.93	3.20	10.18	9.86
VO(3-NO_2Sal-ethanolamine)	Green	$VC_9H_8N_2O_5$	39.29	39.45	2.93	3.20	10.18	9.85
VO(5-MeO-Sal-ethanolamine)	Yellow-green	$VC_{10}H_{11}NO_4$	46.17	45.99	4.26	4.28	5.39	5.43
VO(3-MeO-Sal-ethanolamine)	Gray	$VC_{10}H_{11}NO_4$	46.17	45.97	4.26	4.18	5.39	5.42
VO(Sal-propanolamine)	Green	$VC_{10}H_{11}NO_3$	49.20	49.84	4.54	4.56	5.74	5.44
VO(5-Cl-Sal-propanolamine)	Green	$VC_{10}H_{10}NO_3Cl$	43.11	43.94	3.62	3.90	5.03	4.87
VO(5-Br-Sal-propanolamine)	Green	$VC_{10}H_{10}NO_3Br$	37.18	37.42	3.12	3.39	4.34	4.38
VO(5-NO_2-Sal-propanolamine)	Yellow	$VC_{10}H_{10}N_2O_5$	40.15	41.09	3.37	3.57	9.36	9.62
VO(3-NO_2-Sal-propanolamine)	Green	$VC_{10}H_{10}N_2O_5$	40.15	41.20	3.37	3.62	9.36	9.57
VO(5-MeO-Sal-propanolamine)	Green	$VC_{11}H_{13}NO_4$	48.19	48.67	4.78	4.83	5.11	5.16
VO(3-MeO-Sal-propanolamine)	Green	$VC_{11}H_{13}NO_4$	48.19	48.63	4.78	4.49	5.11	5.27

Table II. Magnetic Susceptibility Data of VO(X-Sal-alcoholamine) Complexes[a]

		Temp, °K						
Complex		78	120	152	189	228	265	Room Temp
VO(Sal-ethanolamine)	χ_M^{cor}	383	821	939	948	934	990	844 (296°)
	μ_{eff}, BM	0.49	0.89	1.14	1.20	1.31	1.37	1.41
VO(5-Cl-Sal-ethanolamine)	χ_M^{cor}		182	366	517	588	631	669 (295°)
	μ_{eff}, BM		0.42	0.67	0.88	1.04	1.16	1.26
VO(5-Br-Sal-ethanolamine)	χ_M^{cor}		202	394	533	597	655	686 (294°)
	μ_{eff}, BM		0.44	0.69	0.90	1.04	1.18	1.27
VO(5-NO_2-Sal-ethanolamine)	χ_M^{cor}		244	396	484	530	580	597 (294°)
	μ_{eff}, BM		0.48	0.69	0.86	0.98	1.11	1.19
VO(3-NO_2-Sal-ethanolamine)	χ_M^{cor}			110	176	238	333	373 (293°)
	μ_{eff}, BM			0.37	0.52	0.66	0.84	0.94
VO(5-MeO-Sal-ethanolamine)	χ_M^{cor}		105	206	305	388	509	578 (295°)
	μ_{eff}, BM		0.32	0.50	0.68	0.84	1.04	1.17
VO(3-MeO-Sal-ethanolamine)	χ_M^{cor}			102	123	190	283	336 (297°)
	μ_{eff}, BM			0.35	0.43	0.59	0.78	0.89
VO(Sal-propanolamine)	χ_M^{cor}	1479	1543	1485	1317	1167	1077	985 (296°)
	μ_{eff}, BM	0.98	1.22	1.34	1.41	1.46	1.51	1.53
VO(5-Cl-Sal-propanolamine)	χ_M^{cor}	1223	1348	1375	1255	1130	1046	982 (294°)
	μ_{eff}, BM	0.87	1.14	1.29	1.38	1.44	1.49	1.52
VO(5-Br-Sal-propanolamine)	χ_M^{cor}	1171	1304	1310	1175	1067	993	938 (293°)
	μ_{eff}, BM	0.86	1.12	1.26	1.34	1.40	1.45	1.48
VO(5-NO_2-Sal-propanolamine)	χ_M^{cor}		301	481	587	660	732	769 (293°)
	μ_{eff}, BM		0.54	0.77	0.94	1.10	1.25	1.34
VO(3-NO_2-Sal-propanolamine)	χ_M^{cor}	1183	1355	1407	1290	1162	1075	1006 (293°)
	μ_{eff}, BM	0.86	1.14	1.31	1.40	1.46	1.51	1.54
VO(5-MeO-Sal-propanolamine)	χ_M^{cor}	1884	1745	1641	1426	1268	1142	1053 (294°)
	μ_{eff}, BM	1.08	1.30	1.42	1.47	1.52	1.56	1.58
VO(3-MeO-Sal-propanolamine)	χ_M^{cor}	1346	1426	1457	1326	1190	1079	1004 (294°)
	μ_{eff}, BM	0.92	1.17	1.33	1.42	1.47	1.51	1.54

[a] Magnetic susceptibilities are in 10^{-6} cgsu. TIP = 50×10^{-6} cgsu. Magnetic moment was calculated using the Curie equation: $\mu_{eff} = 2.84(\chi_M^{cor}T)^{1/2}$ BM. Sal represents salicylaldehyde.

ment. Both Nujol mull and KBr disk techniques were employed. Each spectrum was calibrated with a polystyrene film. Solid-state electronic spectra were recorded in Nujol mull on a Cary 14 recording spectrophotometer.

Analyses. Carbon, hydrogen, and nitrogen analyses were obtained from Galbraith Laboratories Inc., Knoxville, Tenn., and C. F. Geiger, Ontario, Calif. The analytical data are presented in Table I.

General Method of Synthesis of the Complexes. Vanadyl dichloride (0.025 mol) was dissolved in 30 ml of absolute ethanol. Anhydrous sodium acetate (0.05 mol) was added and the mixture was stirred a few minutes and then filtered. Separately, salicylaldehyde or substituted salicylaldehyde (0.025 mol) and the appropriate amino alcohol (0.025 mol) were mixed in 100 ml of absolute ethanol and refluxed for 30 min. The resulting yellow solution of the Schiff base was added slowly to the magnetically stirred filtrate from above containing vanadyl acetate. This mixture was then refluxed for 4 hr while stirring. The separated precipitates were collected on a filter, washed several times with absolute ethanol, and dried in a vacuum desiccator at 30°. The yield is about 65%. The complexes are brown or light green powders which are insoluble in common solvents. The melting points of all the complexes in sealed tubes are greater than 250°.

Results and Discussion

The magnetic susceptibilities and magnetic moments of the complexes are presented in Table II. The room-temperature magnetic moments of the complexes of the X-Sal-ethanolamine series are in the range 0.89–1.41 BM and of the complexes of the X-Sal-propanolamine series are in the range 1.34–1.58 BM. Evidently the room-temperature magnetic moments of the complexes of the X-Sal-propanolamine series are higher than the complexes of X-Sal-ethanolamine. These magnetic moment values are lower than the spin-only value (1.73 BM) expected for a d^1 system and suggest that the complexes are involved in magnetic exchange. The magnetic moments of these complexes decreased considerably as the temperature was lowered (see Table I). The dependence of the magnetic susceptibilities on temperature for both series of complexes is characteristic of antiferromagnetic exchange interaction. The exchange integral J was calculated using the Bleaney and Bowers equation.[9] The g_{av} values for three of our complexes [VO(Sal-ethanolamine), VO(Sal-propanolamine), and VO(5-Br-Sal-ethanolamine)] were obtained from

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Table III. Exchange Integral J Values of the VO(X-Sal-alcoholamine) Complexes[a]

Complex	Exchange integral, J, cm^{-1}
VO(Sal-ethanolamine)	215
VO(5-Cl-Sal-ethanolamine)	330
VO(5-Br-Sal-ethanolamine)	321
VO(5-NO_2-Sal-ethanolamine)	339
VO(3-NO_2-Sal-ethanolamine)	486
VO(5-MeO-Sal-ethanolamine)	398
VO(3-MeO-Sal-ethanolamine)	519
VO(Sal-propanolamine)	141
VO(5-Cl-Sal-propanolamine)	153
VO(5-Br-Sal-propanolamine)	166
VO(5-NO_2-Sal-propanolamine)	294
VO(3-NO_2-Sal-propanolamine)	147
VO(5-MeO-Sal-propanolamine)	117
VO(3-MeO-Sal-propanolamine)	137

[a] $g_{av} = 1.99$; TIP = 50 × 10^{-6} cgsu.

Table IV. Infrared Absorption Frequencies of V=O for the VO(X-Sal-alcoholamine) Complexes

Complex	Freq, cm^{-1}
VO(Sal-ethanolamine)	971
VO(5-Cl-Sal-ethanolamine)	995
VO(5-Br-Sal-ethanolamine)	987
VO(5-NO_2-Sal-ethanolamine)	916
VO(3-NO_2-Sal-ethanolamine)	995
VO(5-MeO-Sal-ethanolamine)	998
VO(3-MeO-Sal-ethanolamine)	976, 995
VO(Sal-propanolamine)	970
VO(5-Cl-Sal-propanolamine)	980
VO(5-Br-Sal-propanolamine)	968
VO(5-NO_2-Sal-propanolamine)	880
VO(3-NO_2-Sal-propanolamine)	963
VO(5-MeO-Sal-propanolamine)	963
VO(3-MeO-Sal-propanolamine)	974, 986

esr measurements. The g_{av} values for these three complexes are the same, 1.99 ± 0.01. The J values of the complexes are presented in Table III. The dependence of the J values on n is quite marked. The J values of the $n = 2$ complexes are higher than the J values for the $n = 3$ complexes. This suggests that the antiferromagnetic exchange interaction is stronger for the VO(X-Sal-ethanolamine) complexes than for the VO(X-Sal-propanolamine) complexes. This difference may be due to the difference in the size of the chelate ring. In the absence of structural data very little can be said about the dependence of the J values on the substituent group. For VO(X-Sal-ethanolamine) complexes, the ligands substituted in the 3 position have larger J values than the complexes of the ligands substituted in the 5 position. For VO(X-Sal-propanolamine) complexes, J values are not so susceptible to substituents with the exception of the 5-NO_2 derivative. Ginsberg, *et al.*, also noticed that J values were higher for their nitro complexes.[2] They also noticed that J values of their complexes are relatively unaffected by the substituent effects.[2]

As the complexes are insoluble in common solvents, esr spectra of the complexes were recorded in polycrystalline samples. From the room-temperature powder spectra g_{av} values could be obtained. Room-temperature spectra of the three complexes studied exhibit only two lines at about 1600 and 3380 G. The 1600-G line may be assigned to the half-field line arising out of the $\Delta M_S = 2$ transition. At liquid nitrogen temperature the half-field line around 1600 G could be located in these three complexes. The observation of half-field spectra suggests the presence of the triplet state in these complexes. No hyperfine splittings of the half-field spectra could be observed in polycrystalline samples at liquid nitrogen temperature. The esr spectrum of VO(Sal-propanolamine) at liquid nitrogen temperature exhibits only one line with $g = 1.99 \pm 0.01$ in the high-field spectra and no hyperfine splittings were observed. The other two complexes show hyperfine splittings in the high-field region. The nature of hyperfine splittings in these complexes is very similar to the reported spectra of oxovanadium(IV) tartrate.[10] The esr spectra were analyzed according to standard procedures[10,11] with the spin Hamiltonian

$$\mathcal{H} = \beta[g_{\parallel}H_zS_z + g_{\perp}(H_xS_x + H_yS_y)] + D[S_z^2 - S(S+1)/3] + AS_zI_z + B(S_xI_x + S_yI_y)$$

A and B values could not be determined accurately due to the overlap of parallel and perpendicular lines. The esr parameters of these complexes are as follows.

Compd	$g_{\parallel}$	$g_{\perp}$	g_{av}	D, cm^{-1}
VO(Sal-ethanolamine)	1.92 ± 0.02	2.02 ± 0.02	1.99 ± 0.01	0.053
VO(5-Br-Sal-ethanolamine)	1.94 ± 0.02	2.01 ± 0.02	1.99 ± 0.01	0.049
VO(Sal-propanolamine)			1.99 ± 0.01	

It is to be noted that $g_{\perp}$ is greater than $g_{\parallel}$, and D of these complexes is greater than that of oxovanadium(IV) tartrate.[10]

In the infrared spectra of the complexes the characteristic V=O stretching frequencies were found in the region 880-998 cm^{-1} (see Table IV). These bands are usually very strong and broad, and in some cases these bands split into two bands. The splitting of the V=O band may be due to unit cell group splitting or to a crystal-packing effect.[2]

The complexes being insoluble in common solvents, electronic spectra were recorded in Nujol mulls. The spectral bands are presented in Table V. All the complexes studied exhibit four bands around 14,000, 16,000, 19,000, and 25,000 cm^{-1}. In the Sal-ethanolamine series the first two bands are not well developed and appear as shoulders. In the Sal-propanolamine series band I also appears as shoulder with the exception of VO(5-MeO-Sal-propanolamine). In most cases bands III and IV are well developed (see Table V). Spectral bands in other subnormal VO(ONO) and VO(ONS) complexes also appear as shoulders.[5,12] The spectral bands are assigned according to the Vanquickenborne and McGlynn (V-M) scheme[13] for oxovanadium(IV) complexes. They have proposed the following ordering of vanadium d orbitals: $d_{xy} < d_{xz}, d_{yz} < d_{x^2-y^2} < d_{z^2}$. According to the V-M scheme band I and band II are assigned to the $d_{xy} \rightarrow d_{xz}$, d_{yz} and $d_{xy} \rightarrow d_{x^2-y^2}$ transitions, respectively. Band III is assigned to the $d_{xy} \rightarrow d_{z^2}$ transitions. Band IV is more intense than all other bands and is believed to be due to charge-transfer and/or intraligand transitions. It seems interesting to compare the electronic spectra of these subnormal complexes with the spectra of normal oxovanadium(IV) complexes,[14] *viz.*, VO(salicylaldimine)$_2$. The

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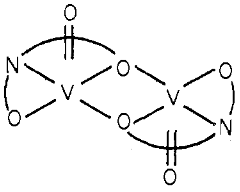
Table V. Electronic Absorption Bands (cm^{-1}) for the VO(X-Sal-alcoholamine) Complexes[a]

Complex	Band I	Band II	Band III	Band IV
VO(Sal-ethanolamine)	14,200 sh	16,600 sh	19,200	26,400
VO(5-Cl-Sal-ethanolamine)	14,000 sh	15,000 sh	19,700	25,300
VO(5-Br-Sal-ethanolamine)	13,400 sh	15,100 sh	19,800	24,400
VO(5-NO_2-Sal-ethanolamine)	12,900 sh	18,200 sh	20,300 sh	27,000
VO(3-NO_2-Sal-ethanolamine)	13,700 sh	16,100 sh	19,000	27,000
VO(5-MeO-Sal-ethanolamine)	13,150 sh	16,900 sh	19,500 sh	24,400
VO(3-MeO-Sal-ethanolamine)	13,700 sh	17,000 sh	19,600	26,600
VO(Sal-propanolamine)	13,700 sh	16,100	18,700	26,400
VO(5-Cl-Sal-propanolamine)	14,200 sh	15,100	18,400	27,000
VO(5-Br-Sal-propanolamine)	13,400 sh	15,100	18,300	26,600
VO(5-NO_2-Sal-propanolamine)	15,000 sh	16,800 sh	19,700 sh	27,400
VO(3-NO_2-Sal-propanolamine)	13,500 sh	16,800	18,900	25,600
VO(5-MeO-Sal-propanolamine)	13,400	16,200	18,500	24,400
VO(3-MeO-Sal-propanolamine)	13,800 sh	16,250	18,700	25,300

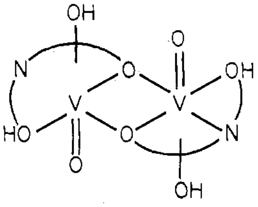
[a] sh = shoulder.

positions of the bands in these two series of complexes do not differ appreciably. The appearance of undeveloped bands is characteristic of electronic spectra of the subnormal oxovanadium(IV) complexes.[5,12]

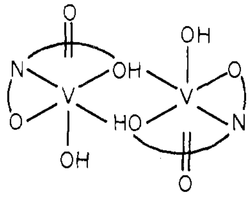
We now wish to comment on the anomaly in the literature of the magnetic moment of these complexes. The magnetic moment of VO(Sal-ethanolamine) is higher than the value reported by Kuge and Yamada[7] but agrees with the work of Poddar, *et al.*[6] The magnetic moments of other two complexes, *viz.*, VO(Sal-propanolamine) and VO(3-MeO-Sal-propanolamine), are also higher than the values reported in ref 7. We synthesized these three complexes by several independent experiments and measured magnetic susceptibilities separately and found deviation in the magnetic moment seldom being greater than ±3%. The infrared spectra of VO(Sal-ethanolamine) and all other complexes do not exhibit any OH stretching vibration and hence these complexes do not contain any water molecule. We suggest structure II for these complexes.[2] Poddar, *et al.*,[6] suggested structure III for their complex without infrared evidence for OH stretching vibration. In III the ligand behaves as a tridentate monobasic ligand. We discard structure III for our complexes on the basis of elemental analysis and infrared data.



II



IIIa



IIIb

Registry No. VO(Sal-ethanolamine), 31320-66-8; VO(5-Cl-Sal-ethanolamine), 36609-87-7; VO(5-Br-Sal-ethanolamine), 36609-88-8; VO(5-NO_2-Sal-ethanolamine), 36609-77-5; VO(3-NO_2-Sal-ethanolamine), 36609-78-6; VO(5-MeO-Sal-ethanolamine), 36609-79-7; VO(3-MeO-Sal-ethanolamine), 36609-80-0; VO(Sal-propanolamine), 31320-67-9; VO(5-Cl-Sal-propanolamine), 36609-81-1; VO(5-Br-Sal-propanolamine), 36609-82-2; VO(5-NO_2-Sal-propanolamine), 36609-83-3; VO(3-NO_2-Sal-propanolamine), 36609-84-4; VO(5-MeO-Sal-propanolamine), 36609-85-5; VO(3-MeO-Sal-propanolamine), 36609-86-6.

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