Table IV (Continued)							
Adsorbent	Contact 5 Gm.	= 10 Min. 20 Gm.	Contact 5 Gm.	= 3 Hrs. 20 Grs.			
р _н 3.0; 1.09	Z soln :	control as					
$(C_{17}H_{23}O_{3}N)_{2}$	H ₂ SO ₄ . J	H₂O; max	. var. =	0.7%			
Talc	93.6			- /0			
Kaolin	93.2	•••		•••			
Silica gel	82.2						
Alumina cream	92.4						
Charcoal	0.6						
$p_{\rm H} 1.0; 1.0?$ (C ₁₇ H ₂₃ O ₃ N) ₂	δ soln.;	control as	say = .96	3.8%			
		$\Pi_2 O$, max	var. =	0.0%			
Talc	93.5	• • •	• • •	• • •			
Kaolin	93.3		• • •	• • •			
Silica gel	80.1	• • •	• • •	• • •			
Alumina cream	93.9		• • •	• • •			
Charcoal	0.4	•••	• • •	• • •			
$p_{\rm H} 5.9; 0.5\%$ (C ₁₇ H ₂₃ O ₃ N) ₂	% soln.; .H₂SO₄.I	control as H ₂ O; max	say = 97 . var. =	$7.2\% \\ 0.4\%$			
Talc	92.6						
Kaolin	91.5						
Silica	81.8						
Alumina cream			• • •	• • •			
Charcoal	0.6						
Table VAdsorption of Cocaine Hydrochloride							
Table V.—Ads							
Adsorbent	Contact 5 Gm.	= 10 Min. 20 Gm.	Contact 5 Gm.	$= \begin{array}{c} 3 \text{ Hrs.} \\ 20 \text{ Gm.} \end{array}$			
Adsorbent Фн 6.7; 1.09	Contact 5 Gm. 6 soln.;	= 10 Min. 20 Gm.	Contact 5 Gm. say = 99	$= \frac{3}{20} \frac{\text{Hrs.}}{\text{Gm.}}$			
Adsorbent Фн 6.7; 1.09	Contact 5 Gm. 6 soln.;	$= 10 \text{ Min.} \\ 20 \text{ Gm.} \\ \text{control as}$	Contact 5 Gm. say = 99	$= \frac{3}{20} \frac{\text{Hrs.}}{\text{Gm.}}$			
Adsorbent $p_{\rm H}$ 6.7; 1.09 $C_{17}H_{21}O_{4}$ Talc Kaolin	Contact 5 Gm. 7 soln.; N.HCl; 93.2 95.3	$= 10 \text{ Min.} \\ 20 \text{ Gm.} \\ \text{control as}$	Contact 5 Gm. say = 99	$= \frac{3}{20} \frac{\text{Hrs.}}{\text{Gm.}}$			
Adsorbent p _H 6.7; 1.09 C ₁₇ H ₂₁ O ₄ I Talc	Contact 5 Gm. 6 soln.; N.HCl; 93.2 95.3 91.3	= 10 Min. 20 Gm. control as: max. var.	$\begin{array}{l} \text{Contact} \\ 5 \text{ Gm.} \\ \text{say} = 99 \\ = 0.7\% \\ \dots \end{array}$	$= \frac{3}{20} \frac{\text{Hrs.}}{\text{Gm.}}$			
Adsorbent $p_{\rm H} 6.7; 1.0\%$ $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream	Contact 5 Gm. 6 soln.; N.HC1; 93.2 95.3 91.3 96.7	= 10 Min. 20 Gm. control as: max. var.	Contact 5 Gm. 5 Gm. 5 ay = 99 = 0.7% 	$= \frac{3}{20} \frac{\text{Hrs.}}{\text{Gm.}}$			
Adsorbent $p_{\rm H}$ 6.7; 1.09 $C_{17}H_{21}O_{4}D_{17}$ Talc Kaolin Silica gel	Contact 5 Gm. 6 soln.; N.HCl; 93.2 95.3 91.3	= 10 Min. 20 Gm. control as: max. var.	Contact 5 Gm. 5 ay = 99 = 0.7% 	$= \frac{3}{20} \frac{\text{Hrs.}}{\text{Gm.}}$			
Adsorbent $p_{\rm H}$ 6.7; 1.0% $C_{17}H_{21}O_{4}T$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0%	Contact 5 Gm. 6 soln.; 93.2 95.3 91.3 96.7 0.2 5 soln.;	= 10 Min. 20 Gm. control as: max. var. 	Contact 5 Gm. 5 Gm. 5 ay = 99 0.7% ay = 10	= 3 Hrs. 20 Gm. 9.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.0% $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$	Contact 5 Gm. 6 soln.; 93.2 95.3 91.3 96.7 0.2 5 soln.; 0.1 0.2 5 soln.;	= 10 Min. 20 Gm. control as: max. var. control as: max. var.	Contact 5 Gm. 5	= 3 Hrs. 20 Gm. 9.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.0% $C_{17}H_{21}O_{4}T$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$ Talc	Contact 5 Gm. 5 Soln.; 93.2 95.3 91.3 96.7 0.2 5 soln.; 0.1 0.2 5 soln.; 93.9	= 10 Min. 20 Gm. control as: max. var. control as: max. var.	Contact 5 Gm. 5	= 3 Hrs. 20 Gm. 9.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.09 $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$ Talc Kaolin	Contact 5 Gm. 5 Gm. 7 soln.; 93.2 95.3 91.3 91.3 91.3 96.7 0.2 5 soln.; 93.9 96.7	= 10 Min. 20 Gm. control as: max. var. control ass max. var.	Contact 5 Gm. 5	= 3 Hrs. 20 Gm. 9.1% 0.1%			
Adsorbent $p_{\rm H} 6.7; 1.0\%$ $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H} 4.6; 1.0\%$ $C_{17}H_{21}O_{4}$ Talc Kaolin Silica gel	Contact 5 Gm. 5 soln.; V.HCl; 93.2 95.3 91.3 96.7 0.2 5 soln.; 0.2 5 soln.; 93.9 96.7 0.2 93.9 96.7 92.1	= 10 Min. 20 Gm. control as: max. var. control as: max. var.	Contact 5 Gm. 5	= 3 Hrs. 20 Gm. 9.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.09 $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$ Talc Kaolin	Contact 5 Gm. 5 soln.; V.HCl; 93.2 95.3 91.3 96.7 0.2 5 soln.; 0.2 5 soln.; 93.9 96.7 0.2 93.9 96.7 92.1	= 10 Min. 20 Gm. control as: max. var. control as: max. var. 	$\begin{array}{l} \text{Contact} & 5 \text{ Gm.} \\ & 5 \text{ Gm.} \\ \text{say} & = 99 \\ & = 0.7\% \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ \text{ay} & = 10 \\ & & & \\ & & & \\ & & & \\ \text{ay} & = 11\% \\ & & & & \\ & & & & \\ & $	= 3 Hrs. 20 Gm. 9.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.0% $C_{17}H_{21}O_{4}D$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 1.0; 1.0%	Contact 5 Gm. 6 soln.; 93.2 95.3 96.7 0.2 9 soln.; 93.9 96.7 92.1 97.9 9.8 90.8 solution:	= 10 Min. 20 Gm. control as: max. var. control as: max. var. control as:	Contact 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 3 gas = 99 0.7% 0.7% 0.7% 1.1% 75.1 86.1 59.6 86.2 0.0 85 gas = 10 85 gas = 10 $85\text$	= 3 Hrs. 20 Gm. 9.1% 0.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.0% $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 1.0; 1.0% s $C_{17}H_{21}O_{4}$	Contact 5 Gm. 6 soln.; 93.2 95.3 96.7 0.2 9 soln.; 93.9 96.7 0.2 9 soln.; 93.9 96.7 92.1 97.9 0.8 solution; N.HCl;	= 10 Min. 20 Gm. control as: max. var. control as: max. var. 	Contact 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 3 gas = 99 0.7% 0.7% 0.7% 1.1% 75.1 86.1 59.6 86.2 0.0 85 gas = 10 85 gas = 10 $85\text$	= 3 Hrs. 20 Gm. 9.1% 0.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.0% $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 1.0; 1.0% s $C_{17}H_{21}O_{4}$ Talc	Contact 5 Gm. 6 soln.; 93.2 95.3 96.7 0.2 9 soln.; 93.9 96.7 92.1 97.9 97.9 90.8 solution; N.HCl; 97.4	= 10 Min. 20 Gm. control as: max. var. control as: max. var. control as: 	Contact 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 3 gas = 99 0.7% 0.7% 0.7% 1.1% 75.1 86.1 59.6 86.2 0.0 85 gas = 10 85 gas = 10 $85\text$	= 3 Hrs. 20 Gm. 9.1% 0.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.0% $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 1.0; 1.0% s $C_{17}H_{21}O_{4}$ Talc Kaolin	Contact 5 Gm. 7 soln.; 93.2 95.3 96.7 0.2 93.9 96.7 0.2 93.9 96.7 92.1 97.9 0.8 solution; N.HCl; 93.9 96.7 92.1 97.9 0.8 solution; N.HCl; 93.9 96.8 solution; 93.9 96.8 solution; 97.9 0.8 solution; 97.9	= 10 Min. 20 Gm. control as: max. var. control as: max. var. control as:	Contact 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 3 gas = 99 0.7% 0.7% 0.7% 1.1% 75.1 86.1 59.6 86.2 0.0 85 gas = 10 85 gas = 10 $85\text$	= 3 Hrs. 20 Gm. 9.1% 0.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.0% $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 1.0; 1.0% s $C_{17}H_{21}O_{4}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 1.0; 1.0% s $C_{17}H_{21}O_{4}$	Contact 5 Gm. 5 soln.; 93.2 95.3 96.7 0.2 5 soln.; o N.HCl; 93.9 96.7 0.2 5 soln.; o N.HCl; 93.9 96.7 92.1 97.9 0.8 solution; N.HCl; 97.9 0.8 solution; 97.4 98.79	= 10 Min. 20 Gm. control as: max. var. control ass max. var. control as max. var. 	Contact 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 3 gas = 99 0.7% 0.7% 0.7% 1.1% 75.1 86.1 59.6 86.2 0.0 85 gas = 10 85 gas = 10 $85\text$	= 3 Hrs. 20 Gm. 9.1% 0.1% 0.1%			
Adsorbent $p_{\rm H}$ 6.7; 1.0% $C_{17}H_{21}O_{41}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 4.6; 1.0% $C_{17}H_{21}O_{4}$ Talc Kaolin Silica gel Alumina cream Charcoal $p_{\rm H}$ 1.0; 1.0% s $C_{17}H_{21}O_{4}$ Talc Kaolin	Contact 5 Gm. 7 soln.; 93.2 95.3 96.7 0.2 93.9 96.7 0.2 93.9 96.7 92.1 97.9 0.8 solution; N.HCl; 93.9 96.7 92.1 97.9 0.8 solution; N.HCl; 93.9 96.8 solution; 93.9 96.8 solution; 97.9 0.8 solution; 97.9	= 10 Min. 20 Gm. control as: max. var. control as: max. var. control as: 	Contact 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 5 Gm. 3 gas = 99 0.7% 0.7% 0.7% 1.1% 75.1 86.1 59.6 86.2 0.0 85 gas = 10 85 gas = 10 $85\text$	= 3 Hrs. 20 Gm. 9.1% 0.1% 0.1%			

SUMMARY

1. The adsorption of codeine, quinine, atropine, pilocarpine and cocaine by talc, kaolin, silica gel, alumina cream and charcoal was investigated.

2. Adsorption of the alkaloids by all five adsorbents was practically unaffected by changes in $p_{\rm H}$ of the original solutions. Adsorption of pilocarpine by silica gel was an exception. In this case a considerably greater per cent of the alkaloid was adsorbed at $p_{\rm H}$ 7.0 than at either $p_{\rm H}$ 1.0 or 4.5, the lowest per cent being adsorbed at $p_{\rm H}$ 4.5.

3. Increasing the period of contact caused a slight increase in adsorption by

silica gel. The adsorption by the other agents remained practically constant.

4. Increasing the weight of adsorbent caused a definite increase in the amount of adsorption of the five alkaloids by all adsorbents. The increase was especially marked in the case of silica gel.

5. Concentration of the solutions had only a slight effect.

6. The affinity of any one adsorbent for the five alkaloids varied only within narrow limits.

7. The adsorption by charcoal was complete or practically complete under all conditions. Adsorption by silica gel was much less than by charcoal, but, in general, greater than by the other adsorbents.

8. Adsorption by talc, kaolin and alumina cream was negligible when 5 Gm. of the adsorbents were used. With 20 Gm. adsorption increased sufficiently to interfere with quantitative recovery.

REFERENCE

(1) Lloyd, J. U., JOUR. A. PH. A., 5 (1916), 381.

The Preparation of Magnesium Silicates by the Interaction of Magnesium Salts and Alkali Metal Silicates*

By R. Roseman, H. Eisenberg and M. B. Levin†

The recent publications of Mutch (1-4), in England, and Levin and co-workers (5, 6), in this country, describing the usefulness of silicates of magnesium in clinical medicine (peptic ulcer therapy), have aroused considerable interest in these materials, as a number of other publications (7) will attest.

The primary purpose of the present paper is to review the literature on the wet reaction between magnesium sulfate (or chloride) and sodium (or potassium) silicate. It is hoped that those interested may thereby gain a better insight into the nature of this

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complex chemical process, and so acquire a more intelligent approach to the problem of preparing various silicates of magnesium for their researches.

The problem is certainly not to be dismissed with the statement, "Alkaline silicates precipitate gelatinous magnesium silicate from magnesium salts; the precipitate is apparently MgO.2SiO₂.2H₂O" (8). And, in the following paragraphs are given, chiefly by means of quotations from the original publications, all available details concerning materials and procedures employed and products obtained, together with observations of interest. Comments of our own are also included.

döbereiner (9)

It is just one hundred years since Döbereiner published his paper, "Analyse und Synthese des Meerschaums." The article is here reproduced in its entirety (Fig. 1).

Döbereiner (9) writes:

"Von einem anderen meiner Zuhörer, Hrn. Gundelach aus Ilmenau, liess ich versuchen, Meerschaum [a hydrous magnesium silicate in which the molecular ratio, MgO:SiO₂ = 2:3] künstlich darzustellen durch Fällung einer sehr verdünnten Auflösung von schwefelsaurer Magnesia mit einer ebenfalls sehr diluirten Solution von kieselsaurem Kali (KO + SiO₃) [2K₂O,3SiO₂?]. Der Versuch gelang vollkommen, wir erhielten einen Niederschlag von kleisterartiger Beschaffenheit, welcher nach oft wiederholtem Auswaschen in einer grossen hohen Kapsel von Saugpapier, an der Luft langsam getrock-

Döbereiner, Analyse d. Meerschaums. 157

ange nicht so angenehm war, als wenn ich einen Stempel gehet hätte, um sie mit erhabenen Figuren auszudrücken. Diem Verfahren bietet den Vortheil dar, dass. wenn man das auglübende Eisen von Neuem darüber bringt, alles ausgelöscht ziel, ohne dass die Substanz beim Schmelzen verbrennt, noch zwei eine Veränderung erleidet, und dass man, da sie von Keen dazu gebraucht werden kann, von einer bestimmten Hinge dieser Zusammensetzung nach und nach bis auf 40 vernkledene Abdrücke mit verschiedenen Stempeln erhalten kann. Wird Jod allein in diesetben Umstände versetzt wie dam Jachlerid, so erzeugt es selbst binnen 14 Tagen keine merkheb Wirkung auf Kork.

Mas könnte diese neue Zusammensetzung Suberon nennen, was dieser Name nicht dem ölartigen Körper gegeben worden wäre, den Boussingault bei der Destillation von korkmeren Kalk erhielt. Uebrigens wird die Elementaranalyse, welche die Chemiker damit anstellen werden, da ich sie selbat nicht vorpelaen konnte, ohne Zweifel einen passenden Namen darbieten, der zugleich die Zusammensetzung und den Ursprung dieser ansen organischen Substanz nusdrückt.

XXV.

Analyse und Synthese des Meerschaums.

Von J. W. DÖBEREINER,

Von Hrn. Eisen ach aus Weimar liess ich in diesem Winim den im Handel vorkommenden orientalischen Meerschaum anhuiren. Derschbe fand darin 1 At. Magnesia, 1 At. Kiesisiure (SiO₃) und 2 At. Wasser, von letzterem also 1 At. mit als Lychnell erhalten. Ich fand dabei, dass der bei der gelindesten Hitze entwässerte Meerschaum sich stark erwirat, wenn man ihn einige Augenblicke in kaltes Wasser inscht, und dass er auch an feuchter Luft sehr bald wieder die gunze Menge des verlornen Hydratwassers ausleht, dass waber durch starkes Glühen die Eigenschaft verliert, nich kin Befeuchten mit Wasser zu erwärmen.

Von einem anderen meiner Zuhörer, Hrn. Gundelneh an Imenau, liess ich versuchen, Meerschaum küustlich darza-

158 Döberginer, Analyse d. Meerschaums.

stellen durch Fällung einer schr verdünnten Auflösung von som felsaurer Magnesia mit einer ehenfalls schr diluirten Solm von kieselsaurem Kali (K $0 + SiO_3$). Der Versuch gin vollkommen, wir erhielten einen Niederschlag von kleistens ger Beschaffenheit, welcher nach oft wiederholtem Auswam in einer grossen hohen Kapsel von Saugpapier, an der is langsam getrocknet, eine leichte, fest zusammenhängende scha baro Masse darstellte. Hr. Gund el a ch im Begriff, a Versuch schr im Grossen zu wiederholen, um zu erfahren a berechnen zu können, ob die Fabricsion des Meerschams i vortheilbaftes Unternehmen werden könne.

Reibt man den natürlichen oder küntlichen Meerschaung haltend mit destillirtem Wasser, so verwandelt er sich is a stelfe kleisterartige Masse. Vermengt man diese Masse min Platinsalmisk, so erhält man einen gelben Telg, woraus bie Kugeln, Schälchen, Tiegelchen u. s. w. geformt werden in nen. Trocknet man diese geformten Massen langsam aus n erhitzt dann dieselben nach und nach bis zum Glüben und lange als sich noch riechende Dampfe estwickeln, so wer sie schwarzgrau, ohne ihre Form zu verlieren. Diese gehn ten Massen werden fast augenblicklich glühend, wenn mas unter dem Zutritte der Luft einem Strome von Wasserstoff ausseizt, bestimmen den Alkohol, sich zu oxydiren, wobei wenn sie noch kein hygroskopisches Wasser enthalten, glübe werden, und wirken auf gewöhnliches Knallgas, d. h. auf d Mischung von 2 Vol. atmosphärischer Luft und 1 Vol. Wa serstoffgas, bei weitem energischer als meine mit Töpfertbonk reiteten eudiometrischen Platinpillen. Besonders überrasch ist es zu schen, wie ein auf den Mittelpunct eines meersch menen Platinschälchens geleiteter dünner Strom von Hydrogi gas diesen Punct plötzlich hellglühend macht, während übrige Oberfläche dunkel oder finster bleibt.

Fig. 1.-The Hundred-Year Old Publication of Döbereiner, J. prakt. Chem., 17 (1839) 157.

net, eine leichte, fest zusammenhängende schneidbare Masse darstellte."

Lefort (10)

Lefort, in his "Mémoire sur les silicates," writes:

"Le silicate de potasse et le sulfate de magnésie donnent lieu à un abondant précipité blanc qui a la plus grande resemblance avec l'hydrocarbonate de magnésie. Ce sel a pour composition: 3(SiO³),-MgO,2HO." (The precipitate was dried at 150– 200°.)

Lefort believed that "une condition indispensable pour obtenir des silicates terreux ayant une composition définie, consiste à opérer avec des solutions étendues," and that "il est de toute nécessité que les sels terreux qui doivent être employés à la préparation des silicates terreux soient parfaitement neutres." Furthermore, the precipitated silicates "doivent être lavés avec soin et avec de l'eau distillée bouillie froide afin de les obtenir purs."

Lefort gives "3(SiO³),KO,9HO" as the potassium silicate employed in his researches.

At the time of the publication of the "Mémoire," the following atomic weights were apparently in use: H = 1, O = 8, Mg = 12, Si = 21 and K = 39.1 (see Kopp-Will's "Jahresbericht," 1861, p. xxi). It would seem, then, that in Lefort's magnesium (or potassium) silicate, the molecular ratio, MgO (or K₂O):SiO₂, was 1:4.5.

von ammon (11)

Although the Library of Congress, Washington, D. C., could find no record of a copy of Von Ammon's work in the United States, summaries appear in Kopp-Will's "Jahresbericht" (11) and Gmelin-Kraut's "Handbuch" (11), stating, in part:

"Der Weg, welchen Ammon zur Darstellung der Silicate der Erden einschlug, bestand darin, dass Er die meist verdünnte Lösung eines Erdsalzes mit der ebenfalls verdünnten des kiesels. Natronsnie derschlug."

"MgSiO₃.—Durch Doppelzersetzung von MgSO₄ mit überschüssigem kristallisiertem Natriumsilikat. --Weisse, sehr voluminöse Gallerte, nach dem Trocknen bald pulvrig, bald dicht und opalartig." (The sodium silicate employed was apparently Na₂SiO₃.8H₂O.)

Two sets of analyses are given, as follows:

	(a)	(b)
Per cent MgO	29.04	29.84
Per cent SiO ₂	46.95	47.31
Per cent H ₂ O	22.54	22.58

The molecular ratios, MgO:SiO₂, are 1:1.09 and 1:1.06 (average = 1:1.08).

Heldt (12)

According to the Heldt publication, "Wird eine Auflösung von Bittersalz mit Wasserglas [apparently $K_2O,3SiO_2$] versetzt, so fällt sogleich ein weisser, gelatinöser Niederschlag, der nach 8 tägigem Stehen in der Flüssigkeit nicht krystallinisch wurde." After drying at 100° , the material was analyzed, yielding 17.0 per cent MgO, 71.4 per cent SiO₂, and 12.7 per cent H₂O. Here, the molecular ratio, MgO:SiO₂ = 1:2.82, so that Heldt's product was not the "single compound, MgO.2SiO₂.2H₂O," given by Kardos and Joffe (19).

"Es wurden nun noch basische Silicate der Magnesia gefällt, indem vorher die Wasserglaslösung mit verschiedenen Mengen Kalilösung versetzt wurde, * * *."

HAUSHOFER (13)

Included in a group of synthetic silicates obtained by Haushofer was a material containing 14.4 per cent MgO, 68.2 per cent SiO₂ and 17.2 per cent H₂O, which was prepared by the action of "Kaliwasserglas" on magnesium sulfate. "Die Niederschläge wurden nach längerem Stehenlassen gut ausgewaschen, mit destillirtem Wasser mehrmals ausgekocht und decantirt, endlich filtrirt; es war meine Absicht, auf diese Weise mechanisch anhaftendes Kali möglichst vollständig zu entfernen."

Haushofer closes his communication with the statement: "Ich vermuthe, dass bei allen diesen Fällungen freie Kieselerde oder ein Hydrat von Kieselerde ausfällt; es ist jedoch sehr schwer, darüber Gewissheit zu erhalten."

It should be pointed out that Haushofer's product, of molecular ratio, $MgO:SiO_2 = 1:3.18$, was not the " $Mg_2Si_7O_{16}.2H_2O$," given by Mutch (1).

MAY (14)

May's technique for preparing a number of silicates follows:

"Die Auflösungen der Metallsalze wurden so lange in die Lösung des kieselsauren Natrons getropft, als noch ein Niederschlag entstand. Die Niederschläge wurden sorgsam ausgewaschen, bei 100° getrocknet * * *."

By proceeding in the above manner, and employing (a) a magnesium sulfate solution of specific gravity 1.052, and (b) a sodium silicate solution of molecular ratio, Na₂O:SiO₂ = 1:1.00, and specific gravity 1.079, May obtained a product containing 28.6 per cent MgO, 49.4 per cent SiO₂ and 22.4 per cent H₂O. ("In der von der Magnesia abfiltrirten Flüssigkeit fand ich kein Alkali.") In this substance, the molecular ratio, MgO:SiO₂ = 1:1.16.

GLASS(15)

Glass states that "if to any of these materials [silicates of soda] an excess of magnesium sulfate solution be added it will be found that all the silica is thrown out of solution together with, roughly speaking, as much magnesium as is the equivalent of the sodium present."

Glass writes further that "the examination of a number of precipitates obtained by the action of magnesium sulfate upon sodium silicate solutions indicated that the primary effect of the addition of a metallic salt was the precipitation of silica; this silica then combined with, or absorbed, or occluded, a certain amount of magnesia, and the extent of this depends upon the opportunity for contact and reaction."

Table I.—Analyses of Glass's Preparations

Experiment	Per Cent MgOª	Per Cent SiO2ª	Molecular Ratio, MgO:SiO2
1	20.54	78.50	$1\!:\!2.57$
2	20.42	78.96	1:2.60
3	30.78	68.64	1:1.50
4	30.57	69.61	1:1.53
5	24.95	74.85	1:2.01
6	24.20	75.80	1:2.10
7	29.15	69.31	1:1.60

^a Ignited basis.

The solutions of sodium silicate used in Experiments 1 and 2 were made from a "crystalline compound" of molecular ratio, $Na_2O:SiO_2 = 1:2.62$ (23.1 per cent Na_2O , 58.6 per cent SiO_2).

The precipitate of Experiment 1 was prepared "from equal weights of the silicate and magnesium sulfate, the magnesium sulfate solution being slowly run into that of the silicate." The ratio, moles $Na_2O_{2.62SiO_2/moles} MgSO_{4.7H_2O}$, was then 0.92.

In Experiment 2, "nine parts of magnesium sulfate were used to ten parts of silicate, and, after precipitation, the whole was evaporated to dryness, ground up and washed." Here, the ratio, moles Na₂O,2.62SiO₂/ moles MgSO₄.7H₂O, was 1.02.

Experiment 3 was a repetition of Experiment 2, but enough caustic soda was added to the Na₂O,-2.62SiO₂ to give Na₂O,1.50SiO₂, and equimolecular amounts of the resulting Na₂O,1.50SiO₂ and MgSO₄.-7H₂O were employed. The object of this experiment, as well as of Experiments 4 and 7, was to prepare a synthetic silicate of molecular ratio, MgO: SiO₂ = 1:1.50 (2:3).

Experiment 4 was the same as Experiment 3 "except that the precipitate was washed straight away instead of first evaporating to dryness."

A "similar series of experiments" was made using a liquid sodium silicate of molecular ratio, $Na_2O:SiO_2 = 1:2.06$ (36.6 per cent SiO₂). Experiments 5 and 6

ROSEMAN AND CO-WORKERS (16)

"The primary purpose of the present investigation was to ascertain the extent of the variation in chemical composition of the products obtained when varying proportions of sodium silicate and magnesium sulfate are allowed to interact."

Га	ble	II	-Résume	é of	Experiments
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Experi- ment ^a	Moles Na2O,3.27SiO2 Moles MgSO4.7H2O	Per Cent MgO, H2O- Free Basis	Per Cent SiO ₂ , H ₂ O- Free Basi3 ^b	Molecular Ratio, MgO:SiO2
	(0.55)	16.59	82.55	1:3.34
`Ē'Ē{2	(1.11)	16.12	82.70	1:3.44
$ \begin{array}{c} Preliminary \\ Series \\ & \\ & \\ & \\ & \\ & \\ & \\ & \\ & \\ & \\ $	(1.66)	13.38	84.44	1:4.24
s (4	0.51	17.80	81.62	1:3.08
Series	0.76	17.39	82.06	1:3.17
<u>∽</u> {6	1,02	16.82	82.42	1:3,29
Final 2	1,53	15.00	84.30	1:8.77
Е (8	2.03	13,41	86.03	1:4,31

^a All of the experiments were conducted at room temperature (approximately 24.5° C.).

^b Complete analyses (SiO₂, MgO, H₂O, R₂O₃, CaO, Na₂O and SO₃) are given in the original publication.

In the final series of experiments (4, 5, 6, 7 and 8, Table II), varying amounts of a 1:3.13 (by volume) water solution of sodium silicate of molecular ratio, Na₂O:SiO₂ = 1:3.27 (8.86 per cent Na₂O, 28.06 per cent SiO₂, specific gravity = 1.384 at 25° C.),¹ were added to *fixed* quantities of a 1:30 (by weight) water solution of Baker's C.P. MgSO₄.7H₂O, the proportions employed being given in column 2, Table II. The sodium silicate solutions were added slowly (5-20 minutes) from a graduate, with stirring (by motor), stirring being then continued for an additional period (5 minutes) to insure thorough mixing. The precipitates were purified by long washing with distilled water, then dried, powdered and analyzed.

But for the use of tap water in the preliminary series of experiments (1, 2 and 3, Table II), the technique employed in this series was essentially that of the final series.

Table IIINomenclature	of	the	Silicic	Acids	and	Silicates
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Ty Ortho- Meta-(proto-) Meso-(deutero-) Para-(tritero-) Tetrero-	$\begin{array}{c} \mathbf{H}_{2n+2}\mathrm{Si}_{n}\mathbf{O}_{3n+1}\\ \mathbf{H}_{2n}\mathrm{Si}_{n}\mathbf{O}_{3n} \end{array}$	1 Mono- H4SiO4 H2SiO3 	$2 \text{ Di-} \\ H_6 \text{Si}_2 \text{O}_7 \\ (H_4 \text{Si}_2 \text{O}_6) \\ H_2 \text{Si}_2 \text{O}_5 \\ \dots \\ \dots$	$3 \text{ Tri-} \\ H_8 \text{Si}_3 \text{O}_{10} \\ (H_6 \text{Si}_3 \text{O}_9) \\ H_4 \text{Si}_2 \text{O}_8 \\ H_2 \text{Si}_3 \text{O}_7 \\ \dots$	4 Tetra- H ₁₀ Si ₄ O ₁₃ (H ₈ Si ₄ O ₁₂) H ₆ Si ₄ O ₁₁ H ₄ Si ₄ O ₁₀ H ₂ Si ₄ O ₉	5 Penta- H ₁₂ Si ₅ O ₁₆ (H ₁₀ Si ₅ O ₁₅) H ₈ Si ₅ O ₁₄ H ₆ Si ₅ O ₁₃ H ₄ Si ₅ O ₁₂
Pentero-	$H_{2n-8}Si_nO_{3n-4}$					H ₂ Si ₅ O ₁₁

were made without the addition of caustic soda; in Experiment 7, equimolecular amounts of Na₂O₂-1.50SiO₂-prepared by the addition of the requisite amount of NaOH to the Na₂O₂O₂O6SiO₂-and Mg-SO₄.7H₂O were employed.

Mutch (4) writes that "Glass's studies are deprived of their scientific interest by his use of inaccurate methods of analysis." Yet, in the same paper (4), Mutch's own analyses of various brands of "magnesium trisilicate" are seen to result in summations ranging from 97.18 per cent to 107.03 per cent. In column 2, Table II, "the ratios given in parentheses were obtained by assuming that the values actually found for the specific gravity $(1.384, 25^{\circ} \text{ C}.)$

¹ The sodium silicate was "E" Brand of the Philadelphia Quartz Company. "The manufacturers of this material state that it is a 40° Baumé liquid holding, according to a typical analysis, 8.70 per cent Na₂O, 28.06 per cent SiO₂, 0.079 per cent Al₂O₃, 0.015 per cent Fe₂O₃, 0.036 per cent TiO₂, 0.020 per cent CaO, 0.042 per cent MgO, 0.035 per cent Cl, 0.025 per cent SO₃, 0.038 per cent CO₂ (molecular ratio, Na₂O:SiO₂ = 1:3.33)."

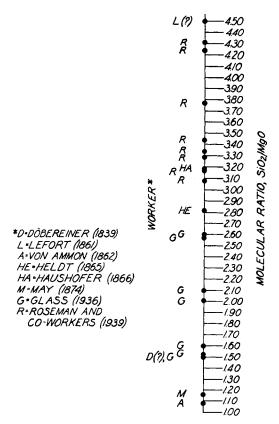


Fig. 2.—Magnesium Silicates Prepared by Various Workers.

and the Na₂O and SiO₂ contents (8.86 per cent and 28.06 per cent, respectively) of the sample of 'E' Brand sodium silicate used in the final series of experiments were applicable to the previously purchased and unanalyzed sample of 'E' Brand employed in the preliminary series of experiments."

From the data obtained in the final series of experiments, it is seen that "with increasing ratios of the amounts of reacting substances, moles Na₂O,- $3.27SiO_2/moles$ MgSO₄.7H₂O, the products show progressively increasing silica contents and correspondingly decreasing magnesia contents (water-free basis), with the attendant increasing ratios, moles SiO₂/moles MgO."

Furthermore, interpolation of these data will show that "a synthetic magnesium silicate, in which the molecular ratio, $MgO:SiO_2$, is the same as the molecular ratio, $Na_2O:SiO_2$, in the sodium silicate used in its preparation (1:3.27), results from the interaction of equal numbers of moles of the sodium silicate and magnesium sulfate."

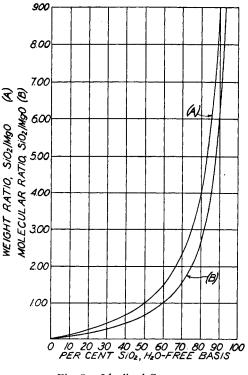
The noteworthy amounts of calcium found in the products of Experiments 1, 2 and 3 (0.29, 0.26 and 0.96 per cent CaO, respectively), more especially Experiment 3, "may be derived from the tap water used in these experiments by base-exchange, by adsorption, and as the result of contamination by precipitated calcium silicate coming from the interaction of dissolved calcium compounds and sodium silicate."

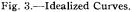
The average Na₂O and SO₃ contents (for the products of Experiments 1–8) being 0.03 per cent and 0.01 per cent, respectively, it was concluded that "thorough washing of the precipitated magnesium silicates enables one to obtain preparations that are essentially free of sodium (and sulfate), even where a considerable excess of sodium silicate has been employed as precipitant."

OTHER WORK

Britton (17), in his "Electrometric Study of the Precipitation of Silicates," found, among other things, that "the precipitates obtained by the interaction of a solution of sodium silicate, Na₂O,2.16-SiO₂, and metallic-salt solutions, except that of calcium chloride, contained less silica than that required to form the metasilicates." (The sodium silicate solution, which was 0.1020N with respect to sodium hydroxide, was added to the magnesium sulfate solution, which was 0.02M.)

Joffe and co-workers (18, 19) thought it desirable, "in view of the conflicting character of the available data, * * * to investigate the nature of the compounds of the alkaline earths with silica from the standpoint of stability and ionic affinity with careful control of the $p_{\rm H}$ of the reacting medium." For a thorough understanding of this excellent work, the reader, preferably familiar with the methods of soil chemistry, is referred to the original publications.





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Finally, a number of additional contributions (20-45) are included in the bibliography, by reason of their pertinence or other interest.

NOMENCLATURE

Concerning nomenclature, Mellor (46) writes:

"The silicates can be regarded as salts of silicic acid and its various condensation products. The general formula of all the silicic acids is $* * * H_{2m}Si_n$ - $O_{2n} + m$, where *n* denotes the number of silicic atoms and *m*, half of the hydrogen atoms per mol. The silicic acids can be named by starting from the *orthosilicic acids*, the acids formed by the loss of one mol of water per mol of acid can be called *protosilicic acids* or, as is more usually the case, *metasilicic acids*; the loss of two mols of water gives *deuterosilicic acids*; the loss of two mols of water gives *deuterosilicic acids*; the loss of four mols of water gives the *terterosilicic acids*; the loss of four mols of water gives the *tetrerosilicic acids* and the loss of five mols of water gives the *penterosilicic acids*."

These acids are illustrated in Table III (Mellor's Table X, with our corrections), "which can be extended to the right and downward if occasion should arise. Each member of a horizontal row differs from the one which precedes by the addition of H_2 -SiO₃; and each member of a vertical column differs from the one which precedes by the removal of H_2 O. The various metasilicic acids are polymers of one another; and those formulas enclosed by brackets in the table are polymers of some other member of the series."

Unfortunately, there is a lack of agreement in the naming of these materials. For example, according to Mellor's classification, $H_4Si_2O_6$ would be metadisilicic (or protodisilicic) acid, and $H_2Si_2O_5$ would be mesodisilicic (or deuterodisilicic) acid. Clarke (47), however, would call the former compound dimetasilicic acid, and the latter, metadisilicic acid. Further, Hildebrand (48) writes that "there are a number of poly-silicic acids, which require such distinguishing terms as di-meta-silicic acid for $H_2Si_2O_5$; tri-meta-silicic acid for $H_2Si_3O_7$; di-ortho-silicic acid for $H_4Si_3O_8$, ctc.," in disagreement with both Mellor and Clarke.

CONCLUSION

The magnesium silicates, prepared by various workers as described in this paper, are conveniently represented as shown in Fig. 2, which is self-explanatory.

In addition, it has been thought desirable to include in this paper Fig. 3, comprising two idealized curves which show how, in a series of pure synthetic silicates of magnesium, the SiO_2 percentages, on a water-free basis, and the corresponding ratios, SiO_2/MgO , are related, weight ratios being the ordinates in Curve (A), while molecular ratios are the ordinates in Curve (B). The curves are portions of equilateral hyperbolas.

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A Study of Ephedrine with Silver Preparations*

By D. J. McLeod and H. G. DeKay[†]

Ephedrine, one of the alkaloids of Ma Huang, has been known and used in medicine for centuries. The plant from which the alkaloid is obtained is Ephedra equisetina, Bunge and Ephedra sinica commonly known as Ma Huang. The active principle, ephedrine, was isolated in 1885 by Yamanashi, in an impure form. Nagai, with the assistance of Hori (1), in 1887, isolated ephedrine in the pure form.

The chemical behavior of ephedrine has been exhaustively studied, and the litera-

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