

stoppered weighing bottle and, with cover removed, placed in an oven at 100° C. for one hour to volatilize the ether. The cover is then put on the weighing bottle and it is cooled in a desiccator and weighed. The loss in weight of the drug represents the ether-soluble extractive.

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## Non-Interfering Adsorbents in Alkaloidal Analysis\*

By Estelle Koozin Johnson and L. Wait Rising†

The purpose of this investigation was to find adsorbents that would be applicable in toxicological analysis involving alkaloids. An agent which coagulates protein material and removes pigments and dyes, but which does not adsorb any dissolved alkaloid, would not only facilitate filtration and clarification but would also eliminate digestion with heat and, therefore, would be especially applicable in analyses involving easily decomposable alkaloids.

Since the discovery by Lloyd (1), in 1910, of the affinity of fuller's earth for alkaloids, much work has been done toward finding an adsorbent which would adsorb alkaloids quantitatively. However, no systematic investigations have been made to determine which adsorbents have no affinity for all or some alkaloids and under what conditions the adsorption is at a minimum.

In this investigation, both the alkaloids and the adsorbents were selected so as to represent different chemical types, limiting the choice to the more commonly used alkaloids and adsorbents. The alkaloids were codeine, quinine, pilocarpine, atropine and cocaine. The adsorbents were talc, kaolin, prime silica gel, alumina cream and Activated Charcoal, U. S. P. XI. Charcoal was included to determine whether it has any selective action whatsoever, and to determine to what extent certain physical conditions affect its adsorptive capacity.

## EXPERIMENTAL

*General Procedure.*—Aqueous solutions of the alkaloidal salts (0.5% and 1.0%) were prepared and the  $p_H$  adjusted to definite values. Five-gram and 20-gram portions of the adsorbents were placed in different bottles and 100-cc. portions of the alkaloidal solution were pipetted into each bottle.

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## NOTICE

Authors who desire to make corrections in papers read at the Richmond meeting, please do so promptly so that the manuscript may be sent to the Board of Review without delay.

Table I.—Adsorption of Codeine Sulfate

Assay of dry salt = 101.8% (C<sub>18</sub>H<sub>21</sub>O<sub>3</sub>N)<sub>2</sub>.H<sub>2</sub>SO<sub>4</sub>

Adsorbent	Contact = 10 Min.		Contact = 3 Hrs.	
	5 Gm.	20 Gm.	5 Gm.	20 Gm.
<i>p</i> <sub>H</sub> 4.9; 1.0% soln.; max. var. = 1.2%				
Talc	97.7	87.0	99.2	87.7
Kaolin	96.7	84.2	98.2	87.4
Silica gel	88.6	61.0	86.8	55.0
Alumina cream	95.7	84.3	95.2	85.7
Charcoal	0.3	1.1	3.1	1.6
<i>p</i> <sub>H</sub> 3.2; 1.0% soln.; max. var. = 0.6%				
Talc	97.7	90.3	97.0	...
Kaolin	97.3	87.5	96.5	...
Silica gel	88.1	56.8	84.0	...
Alumina cream	95.9	88.3	95.3	...
Charcoal	0.3	3.0	0.6	...
<i>p</i> <sub>H</sub> 1.6; 1.0% soln.; max. var. = 0.8%				
Talc	99.4	...	...	...
Kaolin	97.1	...	...	...
Silica gel	86.8	...	...	...
Alumina cream	97.3	...	...	...
Charcoal	1.4	...	...	...
<i>p</i> <sub>H</sub> 7.7; 1.0% soln.; max. var. = 0.2%				
Talc	97.4	...	...	...
Kaolin	97.0	...	...	...
Silica gel	80.8	...	...	...
Alumina cream	95.5	...	...	...
Charcoal	1.5	...	...	...
<i>p</i> <sub>H</sub> 4.8; 0.5% soln.; max. var. = 1.1%				
Talc	95.8	78.4	98.8	...
Kaolin	97.9	76.2	95.7	...
Silica gel	88.2	57.2	85.9	...
Alumina cream	98.2	81.8	97.9	...
Charcoal	3.2	6.2	3.1	...

Table II.—Adsorption of Quinine Dihydrochloride

C<sub>20</sub>H<sub>24</sub>O<sub>2</sub>N<sub>2</sub>.2HCl; max. var. = 1.3%

Adsorbent	Contact = 10 Min.		Contact = 3 Hrs.	
	5 Gm.	20 Gm.	5 Gm.	20 Gm.
<i>p</i> <sub>H</sub> 6.2; 1.0% soln.; control assay = 102.9%				
Talc	92.5	...	...	...
Kaolin	91.6	...	...	...
Silica gel	86.8	...	...	...
Alumina cream	94.1	...	...	...
Charcoal	0.0	...	...	...
<i>p</i> <sub>H</sub> 3.0; 1.0% soln.; control assay = 95.5% C <sub>20</sub> H <sub>24</sub> O <sub>2</sub> N <sub>2</sub> .2HCl; max. var. = 0.6%				
Talc	94.4	84.1	92.7	82.6
Kaolin	94.9	87.1	94.2	85.7
Silica gel	94.6	78.5	91.8	71.4
Alumina cream	94.6	86.1	94.7	86.9
Charcoal	1.2	0.5	0.5	0.0
<i>p</i> <sub>H</sub> 1.0; 1.0% soln.; control assay = 100.1% C <sub>20</sub> H <sub>24</sub> O <sub>2</sub> N <sub>2</sub> .2HCl; max. var. = 0.9%				
Talc	97.4	...	...	...
Kaolin	98.2	...	...	...
Silica gel	91.6	...	...	...
Alumina cream	96.3	...	...	...
Charcoal	0.6	...	...	...
<i>p</i> <sub>H</sub> 3.0; 0.5% soln.; control assay = 100.5% C <sub>20</sub> H <sub>24</sub> O <sub>2</sub> N <sub>2</sub> .2HCl; max. var. = 0.9%				
Talc	92.4	...	...	...
Kaolin	92.7	...	...	...
Silica gel	92.5	...	...	...
Alumina cream	95.7	...	...	...
Charcoal	1.3	...	...	...

The bottles were vigorously shaken on a mechanical shaker for ten minutes and three hours. The mix-

tures were filtered, 25-cc. aliquot portions of the filtrate were pipetted into separatory funnels and the residual alkaloid was extracted by the usual method, using chloroform as the immiscible solvent. The chloroform extracts were evaporated, the residues dried at 100–105° C. and the percentages of recovery were calculated.

*Results.*—The figures in Tables I to V represent the percentages of recovery and are expressed as alkaloidal salt. Codeine sulfate, which contains 5 molecules of water of crystallization, was dried and assayed before being used. Solutions prepared from it were exactly 0.5% or 1.0% in strength, calculated as (C<sub>18</sub>H<sub>21</sub>O<sub>3</sub>N)<sub>2</sub>.H<sub>2</sub>SO<sub>4</sub>. The other alkaloidal salts were used without drying. Solutions were prepared approximately 0.5% and 1.0% in strength and control analyses were conducted. The calculations for both the controls and the adsorption determinations, were based on the assumption that the solutions contained exactly 0.5% or 1.0% of the alkaloidal salt.

Table III.—Adsorption of Pilocarpine Hydrochloride

Adsorbent	Contact = 10 Min.		Contact = 3 Hrs.	
	5 Gm.	20 Gm.	5 Gm.	20 Gm.
<i>p</i> <sub>H</sub> 7.0; 1.0% soln.; control assay = 94.4% C <sub>11</sub> H <sub>16</sub> O <sub>2</sub> N <sub>2</sub> .HCl; max. var. = 1.5%				
Talc	92.5	...	...	...
Kaolin	90.8	...	...	...
Silica gel	64.0	...	...	...
Alumina cream	91.8	...	...	...
Charcoal	0.3	...	...	...
<i>p</i> <sub>H</sub> 4.5; 1.0% soln.; control assay = 96.5% C <sub>11</sub> H <sub>16</sub> O <sub>2</sub> N <sub>2</sub> .HCl; max. var. = 1.3%				
Talc	90.6	87.9	92.4	89.7
Kaolin	92.2	88.2	93.0	90.5
Silica gel	87.7	67.9	86.0	69.5
Alumina cream	91.0	82.6	90.1	84.2
Charcoal	3.4	0.5	4.7	1.2
<i>p</i> <sub>H</sub> 1.0; 1.0% soln.; control assay = 96.7% C <sub>11</sub> H <sub>16</sub> O <sub>2</sub> N <sub>2</sub> .HCl; max. var. = 1.2%				
Talc	93.0	...	...	...
Kaolin	93.7	...	...	...
Silica gel	80.9	...	...	...
Alumina cream	89.9	...	...	...
Charcoal	0.0	...	...	...
<i>p</i> <sub>H</sub> 4.5; 0.5% soln.; control assay = 89.6% C <sub>11</sub> H <sub>16</sub> O <sub>2</sub> N <sub>2</sub> .HCl; max. var. = 0.8%				
Talc	88.6	...	...	...
Kaolin	89.0	...	...	...
Silica gel	84.2	...	...	...
Alumina cream	89.2	...	...	...
Charcoal	0.5	...	...	...

Table IV.—Adsorption of Atropine Sulfate

Adsorbent	Contact = 10 Min.		Contact = 3 Hrs.	
	5 Gm.	20 Gm.	5 Gm.	20 Gm.
<i>p</i> <sub>H</sub> 5.9; 1.0% soln.; control assay = 99.2% (C <sub>17</sub> H <sub>23</sub> O <sub>3</sub> N) <sub>2</sub> .H <sub>2</sub> SO <sub>4</sub> .H <sub>2</sub> O; max. var. = 0.9%				
Talc	96.8	88.5	94.6	85.3
Kaolin	96.5	84.1	94.9	84.0
Silica gel	84.1	54.0	82.3	49.0
Alumina cream	95.2	82.1	93.6	83.2
Charcoal	2.7	0.7	1.3	0.2

Table IV (Continued)

Adsorbent	Contact = 10 Min.		Contact = 3 Hrs.	
	5 Gm.	20 Gm.	5 Gm.	20 Gm.
$p_H$ 3.0; 1.0% soln.; control assay = 96.8% ( $C_{17}H_{23}O_3N$ ) <sub>2</sub> .H <sub>2</sub> SO <sub>4</sub> .H <sub>2</sub> O; max. var. = 0.7%				
Talc	93.6	...	...	...
Kaolin	93.2	...	...	...
Silica gel	82.2	...	...	...
Alumina cream	92.4	...	...	...
Charcoal	0.6	...	...	...
$p_H$ 1.0; 1.0% soln.; control assay = 96.8% ( $C_{17}H_{23}O_3N$ ) <sub>2</sub> .H <sub>2</sub> SO <sub>4</sub> .H <sub>2</sub> O; max. var. = 0.3%				
Talc	93.5	...	...	...
Kaolin	93.3	...	...	...
Silica gel	80.1	...	...	...
Alumina cream	93.9	...	...	...
Charcoal	0.4	...	...	...
$p_H$ 5.9; 0.5% soln.; control assay = 97.2% ( $C_{17}H_{23}O_3N$ ) <sub>2</sub> .H <sub>2</sub> SO <sub>4</sub> .H <sub>2</sub> O; max. var. = 0.4%				
Talc	92.6	...	...	...
Kaolin	91.5	...	...	...
Silica	81.8	...	...	...
Alumina cream	92.7	...	...	...
Charcoal	0.6	...	...	...

Table V.—Adsorption of Cocaine Hydrochloride

Adsorbent	Contact = 10 Min.		Contact = 3 Hrs.	
	5 Gm.	20 Gm.	5 Gm.	20 Gm.
$p_H$ 6.7; 1.0% soln.; control assay = 99.1% $C_{17}H_{21}O_4N$ .HCl; max. var. = 0.7%				
Talc	93.2	...	...	...
Kaolin	95.3	...	...	...
Silica gel	91.3	...	...	...
Alumina cream	96.7	...	...	...
Charcoal	0.2	...	...	...
$p_H$ 4.6; 1.0% soln.; control assay = 100.1% $C_{17}H_{21}O_4N$ .HCl; max. var. = 1.1%				
Talc	93.9	...	75.1	...
Kaolin	96.7	...	86.1	...
Silica gel	92.1	...	59.6	...
Alumina cream	97.9	...	86.2	...
Charcoal	0.8	...	0.0	...
$p_H$ 1.0; 1.0% solution; control assay = 101.4% $C_{17}H_{21}O_4N$ .HCl; max. var. = 1.7%				
Talc	97.4	...	...	...
Kaolin	98.5	...	...	...
Silica gel	87.9	...	...	...
Alumina cream	94.9	...	...	...
Charcoal	0.2	...	...	...

## 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  $p_H$  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  $p_H$  7.0 than at either  $p_H$  1.0 or 4.5, the lowest per cent being adsorbed at  $p_H$  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.

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## 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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