THE BLACK VAULT

AB 7514

COPY

AM, 7, 5/6

September 15, 1951

TO [redacted] C

FROM [redacted]

SUBJECT: Examination of unknown substance contained in metallic collapsible tube.

Exhibits: Three photographic negatives and four 8" x 10" photographic enlargements of the tube and container as submitted.

Two graphs entitled Curve 1 and Curve 2 representing ultraviolet absorption spectra of the unknown material.

Physical Description of the Unknown: See photos.

Sample as submitted consisted of a collapsible metallic tube contained in clear plastic cylinder.

Tube: The tube was about 2" overall and was composed of a body about 1¼" wide at the base, and a neck and tapering nozzle. The tube body was about an inch in length as received. Unrolled it measured about 1½". The neck and nozzle were about 3/4" in length. The appearance of the outer surface of the tube body was dull white and appeared to have been painted. Its surface was soiled. It contained no labelling on its surface. Examination in ultraviolet light revealed no invisible marks or labelling. The tube as received was uncollapsed. The neck and shoulder contained a dried grey-white exudate. The nozzle opening was corroded and its bore was closed up with decomposed solid material.

Cylinder: The clear plastic cylinder is about 1 5/16" in diameter and about 1½" in length. It is divided into two compartments, internally,

separated by a wall. Each end is capped with a precision ground friction cap. The cap of the smaller compartment end contains a minute bore through its center and centered through the wall separating the compartments so that a hypodermic needle may be inserted through the small compartment from the outside into the larger compartment. The rusted fragments of a needle were found in the small compartment, the sides of which were stained with brownish red smears. Scrapings of these smears were taken for possible analysis . They gave strong positive test for iron.

Examination and Analysis: Part 1. September 12-14, 1951

The unknown was opened, physically examined and photographed. The nozzle was penetrated with a hypodermic needle and one droplet of the contents was removed by inversion and gentle pressure on the tube walls and was placed on a microscopic glass well-slide.

The droplet was a greyish white, somewhat viscous liquid suspension. It was odorless and apparently aqueous. Examination under low power microscope showed it to be heavily laden with particulate matter. A number of clusters of cellular matter with greenish cell walls were observed at low power. The particulate matter was insoluble in ethanol. Treatment with dilute sulfuric acid (10%) produced large numbers of crystals of calcium sulfate identified positively in polarized light. Slight warning of the slide produced effervescence.

A second droplet was placed in a cuvette containing 2 ml. of distilled water and an ultraviolet absorption curve was made in the range of 220-350 mu. Samples of barbital and scopolamine were run and curves recorded; they show no absorption characteristics of the unknown. (for curves, see graph entitled curve 1.) The curve of the unknown is not characteristic

of any chemical or pharmaceutical known to the [redacted] H-B13 Laboratory.

Part 2

Analysis - Attempts to remove more of the sample than the 2 droplets, previously noted, were unsuccessful. After completely collapsing the tube, the neck and shoulders were carefully scraped to remove solid exudate. This material was taken up in 1ml of methanol and labelled Sample 1.

The tube itself was cut open with a razor blade revealing a shiny brass colored surface in good state of preservation. Its lower extremity showed a thin coating of a black rubber-like tacky compound. Probably a seal for the folded bottom edge. There was no visible evidence of any material on the surface.

The inner surface of the tube was washed with successive portions of absolute methanol totalling several mililiters and the recovered liquid, a milky liquid, was labelled Sample 2.

Samples 1 and 2 were warmed on a steam bath, cooled to room temperature, centrifuged and ultraviolet spectra were made of each on the Cary Recording Spectrophotometer. Both exhibit absorption in the ultra-violet region of the spectrum but neither curve was characteristic of any chemical or pharmaceutical substance known to the analyst [redacted] H-B16.

The solutions were evaporated to dryness and an attempt made to produce a record of their infra-red spectra but the analyst reported to success for lack of sample.

Concluding Remarks - The analyst has emphasized that although certain data have been presented in this report giving absorption curves, the virtual lack of sample causes him to present these data with reservations since it is not

certain in his view that the scrapings or residual droplets or washings represent the materials desired to be analyzed. The amount of material submitted makes identification impossible.

Needles

1. The needle is designed for injection its gage is not exactly the same as standard domestic need

2 The gage of the needle indicates that it would be used for venipuncture

3. Subject is burn wounded

4. The hub of the needle, which is made of brass, is a carefully produced instrument The needle is too badly decomposed to make a study

5. Yes

6. The needle is very slightly smaller than a 19 gage hypodermic nickle. Its outside diameter is .040" (its present length is .792"). ~~Hypodermic needles generally are produced~~ in 1/4", 3/8", 1/2", 5/8", 3/4", 1" and larger. Since the maximum length the instrument could penetrate would be a 1" needle the skin

whose diameters are usually .042" outside diameter and .027" inside diameter

10. No. in my opinion, and this can be proved by metallographic examination, it is part or hub of the hypodermic need

11. Yes. altho no quicker than an injecti given by syringe. Probably the design was developed to avoid transfer or to disguise the injection. Injection and are generally put up in glass ampu which are transferred to syringes. It is possible that without the needle the injecti in the tube could pass for some other type of medicine. also by this means an injecti could be given without equipment other than a needle. In all it is a crude method of putting up a potent drug, if that is what it wa intended to contain.

The Container

1. no reason apparent to us

2.

3. water proof but could not with
high heat without fusing.
could nt be sterlized by heat for exam

4 dont know.

5.

6 none.

7 none

8 — Pharmacopeial drugs and
in general use in the Physician's armamentaria

proprietary or no
proper proprie
and the
Pharm drug

The Collapsible Tube

1. Aluminum (of spectrogram)

2. It could have with stood reasonable pressure.
   (a) yes
   (b) no
   (c) yes. the tube is not fragile and would not spill its contents unless cut open

3. Difficult to say. They could have been made by hand by a careful technician

4. No.

5. Liquids

6. Yes

7. Fire proof but not heatproof.

8. Probably not.

9. If by this is meant: directions, no.

14. Microscopic examination reveals no hairs. The needle ~~is too~~ and splits clinging to it ~~could~~ did not ~~not be identified as having~~ appear to have any cellular characteristics.

15. No. The manner in which it was supported is ~~unknown to us~~ not apparent.

7. The bore was clogged with ~~(rust & oxide dust)~~ rust. ~~[illegible]~~ Scrapings of rust and decomposed matter in the order of 10-25 mg. from the outside surface of the needle gave a positive test for blood with the Benzidine test.

8+9 This should be investigated. I suggest that an expert of the [redacted] be interviewed H-B/3

10 Generally not this type. ~~[illegible]~~ smallpox typhoid sewing typ 26 gage ne not this kind
~~[illegible] such as used for a [illegible] of sewing~~

11. Probably not ~~She~~ "~~gelatinous~~" would have to ~~be~~ [illegible]
Probably glycerin could be forced thru whereas ~~the~~ thicker gels. This depends on the viscosity of the gel.

12 No.

13 See 8+9.