

Valentine To Valy

Valov

I have  
of June and  
I have nothing to do  
with the selection of  
personnel, all I can  
do is to refer your  
letter to the UN which  
I will do.

ask my attorney

6-19-47

Mrs. Franklin D. Roosevelt  
Representative

December 11, 1946.

Dear Mr. Valov:

I have read with deep sympathy the story of Mrs. Carstens. My heart goes for these unfortunate people, who, because they live, are entitled to happiness and peace. I am sure you will understand that it is humanly impossible for me to consider each case individually since I receive hundreds of letters of appeal similar to yours. That is why I am devoting my time and energy to developing an international program broad enough that all may benefit by it.

The Committee-approved draft Constitution of the International Refugee Organization will be presented to the General Assembly for recommendatory action sometime this week-end. It is to be hoped that the Organization can be established without too much delay. I suggest that you follow subsequent developments in your local newspapers, for this subject will undoubtedly be given wide coverage, in order to advise and assist your friend as soon as legal ways and means are available.

Sincerely yours,



Mrs. Franklin D. Roosevelt

Mr. Paul Valov,  
1951 Thousand Oaks Blvd.,  
Berkeley 7, California.

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# Rapid Qualitative and Quantitative Determination of Barbiturates from Postmortem Specimens

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## Rapid Qualitative and Quantitative Determination of Barbiturates from Postmortem Specimens

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**A** METHOD is described for the separation of barbiturates from postmortem specimens by extraction with aqueous sodium hydroxide. Proteins are coagulated with sodium tungstate upon acidification with sulfuric acid and, following filtration, the filtrate is extracted with ether. Evaporation yields the barbiturates usually in crystalline form ready for qualitative and quantitative determination. The yield is complete. The elapsed time required is 90 minutes.

The prevalent use of barbiturate drugs has resulted in a steady increase in the number of postmortem cases in which barbiturate is the issue. The introduction in this laboratory of the method described below has resulted in large savings in time and reagents.

### PROCEDURE FOR EXTRACTION OF BARBITURATE FROM BLOOD AND VISCERA

1. Measure 60 ml. of blood (or minced viscera), 410 ml. of distilled water, and 10 ml. of 10% sodium hydroxide into a 1-liter flask. Shake 5 minutes.

2. Add 60 ml. of 10% sodium tungstate. Add slowly 60 ml. of 0.67 *N* sulfuric acid with continuous shaking, and acidify with 18 *N* sulfuric acid until acid by Universal indicator paper.

3. Filter to collect 450 ml., extract with an equal volume of redistilled ethyl ether, and shake 5 minutes. Separate from the water phase and evaporate.

The relatively large volume of ether prevents emulsions, which occasionally occur with smaller quantities of ether. The evaporation is normally performed in distillation equipment. The ether is recovered and re-used in later extractions. This procedure permits the attainment of lower blanks.

4. Weigh, deduct blank, and calculate milligrams per 100 grams.

Determination of Blanks. Add 250 mg. of phenobarbital to 60 grams of liver and extract according to preceding directions. The average recovery is 253 mg., representing the barbiturate plus blank. Extract in the same way 60 grams of liver. The average residue is 3 mg.

The established average of foreign matter per 60 grams of liver may be subtracted in the quantitative determination of high positive cases. Low positive cases call for a colorimetric quantitative determination of barbiturate.

5. Identify the presence of barbiturate by the customary Zwikker-Koppanyi method.

The Rotondaro (1) method may be employed for final purification of the residue, in which case the color effects with the Zwikker-Koppanyi reagents will be brighter. This is not necessary, however, as the presence of a few milligrams of barbiturate may be established conclusively without Rotondaro purification.

### DISCUSSION

The distinctive feature of this method is the extraction of a small quantity of tissue with sodium hydroxide which brings the barbiturate directly into solution, separating it simultaneously from fats and oils. The coagulation of proteins yields a clear solution, substantially purified, so that the final ether extraction is rapid and complete. The final residue is usually crystalline, ready for qualitative and quantitative determination. Melting points can also be established after sublimation. Secanol residues and, sometimes, nembutal are noncrystalline, but the latter also becomes crystalline after evaporation of added water. In practice, the following residual quantities of ingested barbiturates are encountered: for secanol, up to 3 mg. per 100 grams of liver; for nembutal (pentobarbital), up to 16 mg., and for phenobarbital, up to 39 mg.

Cases are very rare where barbiturate is accompanied by other substances extractable by ether from an acid medium (such as salicylic acid, sedormid, or acetophenetidin). Since therefore any abnormal weight exceeding 40 mg. of ingested barbiturate per 100 grams of tissue would attract the attention of the toxicologist, it could not be overlooked, and would indicate interference of the above type. This makes the method practically specific for commonly used barbiturates.

The advantages of the method become obvious if it be contrasted to the customary method employing ethyl alcohol. Primarily, in order to ensure recovery from low positive barbiturate cases, large quantities of tissue, up to 400 grams, are subjected to four alcohol extractions, followed by evaporation. This procedure, besides being cumbersome, yields a gummy paste, formed by heating the fatty and oily components of human tissue which are extracted with the barbiturate. Subsequent water and ether extractions yield a final noncrystalline residue requiring additional purification. The elapsed time required is from 2.5 to 3 days. Endeavors to reduce the time by working with smaller amounts of tissue and decreasing the number of consecutive alcohol extractions often leads to failure in low positive cases.

### LITERATURE CITED

- (1) Rotondaro, F. A., *J. Assoc. Official Agr. Chem.*, 23, 777-82 (1940).