

THE DEPARTMENT OF THE AMERICAN ASSOCIATION OF COLLEGES OF PHARMACY

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DEPARTMENT.

"I suppose every teacher of analytical chemistry has his own ideas of the content of applied quantitative analysis or drug assay. Since the title of most courses is 'Drug Assay,' naturally the material must be taken from the practical everyday analysis necessary to meet the requirements of the Pure Food and Drugs Act. Some colleges prefer to make this course a part of quantitative analysis, others prefer to give a fundamental course in quantitative analysis followed by one in drug assay. Regardless of which method is pursued, the following paper by Russell L. Taylor will be interesting and instructive to all teachers of such a course."—C. B. JORDAN, *Editor*.

A COURSE IN DRUG ASSAY TO MEET COMMERCIAL REQUIREMENTS.

BY RUSSELL L. TAYLOR.*

I use the term "commercial" to apply to forms of employment requiring pharmaceutical knowledge and skill, without involving the practice of pharmacy as a profession.

The federal, state and city governments, in their various laboratories, need men and women trained in the methods and technic of drug assay. Large companies manufacturing pharmaceutical products require men of like training in their control laboratories and other positions of responsibility. Private laboratories open another door of opportunity to the graduate who has the requisite knowledge and skill to undertake such work. If the pharmacy school does not train men to fill these positions, the chemistry department will do so, and I think you will agree that such training comes more naturally into the province of pharmacy than of chemistry. I believe that it is to the advantage of Pharmacy, as a profession, to have such positions filled by men who have its interests at heart, and who, both by inclination and education, are sympathetic with its problems and its ethical standards.

When we train our student to fill such positions, we increase his opportunities for employment, which is no mean advantage in these times. In addition, for the graduate who prefers the comparative tranquillity of a laboratory to the keen competition of retail merchandising, we provide an avenue of congenial occupation.

In planning the course in Drug Assay offered at the State College of Washington, I have reviewed carefully my own experience, both in a government laboratory and in the control laboratory of a large company, and have tried to incorporate the essential facts which a beginner will be expected to know and the methods in which he will be expected to be proficient when he enters a position in either of these fields. It must be realized that each laboratory has its own special system and requirements for an analyst, and while it is impossible to prepare the student for every specific routine in every laboratory, we can and must see that he is trained in the methods, technic and mental attitude that will enable him to adapt himself quickly to the requirements of any particular situation.

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The time required for this course as given at the State College of Washington is three semester hours, divided into one lecture and six hours of laboratory work each week. It must be given either during the junior or senior year, so that the chemistry requirements will have been fulfilled, and it is included in our senior schedule as a required course.

The student is impressed, first of all, with the importance of absolute neatness in the laboratory. Spilled drugs or chemicals must be wiped up immediately, and desks and equipment left clean at the end of the period. Each member of the class is required to keep a note-book and report his work in specified forms outlined under each preparation to be assayed. Emphasis is laid upon the development of technic, not merely doing the work, but doing it in the best way. The student is expected to learn the theories and procedures and know the reason for every step in the method before beginning any assay. This not only saves his time at the moment, but the habit is very valuable. He is supplied with a syllabus of all laboratory work, and this must be studied in advance of the laboratory period.

The lectures include explanations of methods used in the assay of laboratory samples and other special methods, theory of distribution constant, use of proper indicators, explanation of calculations in all methods, and similar material. Problems are assigned at each period to be solved outside of class. These are based on correction factors, adjusting of fluidextracts and tinctures for alcohol and alkaloid content, calculating conversion factors, and so on. Discussion designed to lead the student to do original thinking in regard to new methods is encouraged and stimulated.

Very few manufacturing houses make only official preparations, and government analysts must work on many unofficial ones, so our course includes both types. The first work done is the making of standard solutions, including $N/10$ succinic acid by weight, $N/10$ KOH, $N/10$ H_2SO_4 and $N/50$ KOH.

The following unknowns are assayed by each student:

1. Fluidextract of belladonna leaves for total alkaloids.
2. Tincture of Nux Vomica for alcohol content and total alkaloids.
3. Tincture of Cinchona for alcohol and total alkaloids.
4. Extract of Nux Vomica for total alkaloids.
5. Pilular extract of stramonium for total alkaloids.
6. Powdered stramonium for total alkaloids.
7. Cinchona bark for total alkaloids.
8. A powder for per cent of quinine, both gravimetric and volumetric methods.
9. Tablets of strychnine sulphate for per cent of strychnine sulphate.
10. Coated pills of quinine sulphate for per cent of quinine sulphate, both gravimetric and volumetric methods.
11. Insoluble powder containing a volatile oil for the per cent of volatile oil.
12. Cough syrup for the amount of chloroform.
13. Tobacco for the per cent of nicotine.
14. Headache powder for the per cent of caffeine and acetanilid.
15. A special unknown.

It will be noticed that the first half of the course deals with official preparations and the last half with unofficial ones.

The first unofficial preparation is a powder to be assayed for the per cent of quinine. This is first determined gravimetrically and then the same residue is

titrated, using brom-cresol purple as indicator. This sample gives the student a comparison between gravimetric and volumetric methods.

The second unofficial preparation is compressed tablets of strychnine sulphate. This is assayed not so much for the practice in assaying, as for the calculation of the conversion factor, to give the student practice in determining strychnine and calculating the sulphate, also for the experience in reporting uniformity of thickness, disintegration time, etc.

The third is coated pills of quinine sulphate for the per cent of quinine sulphate. This is determined first gravimetrically and then volumetrically. This sample again gives the student valuable practice in calculating factors. For example, the sample is weighed as anhydrous quinine and calculated as quinine sulphate, U. S. P., with seven molecules of water. It is titrated as quinine and calculated as quinine sulphate. This preparation also gives practice in taking the sample by the aliquot part method. The pills are first dissolved in a liter of water and an aliquot part is then removed for the determination.

The fourth unofficial sample is an insoluble powder containing a volatile oil, for the determination of the per cent of volatile oil. For this purpose, unknowns are prepared containing talcum powder and any ether-soluble volatile oil. This sample is weighed into a Soxhlet thimble and the oil extracted with ether, the thimble weighed after drying, and the per cent of volatile oil calculated by difference. This sample gives the student experience with the continuous extractor.

The fifth unofficial sample is a cough syrup containing chloroform for the determination of chloroform. The chloroform is converted to sodium chloride and titrated as such with *N*/10 silver nitrate. Here the student gains more experience in making standard solutions and begins to realize that the knowledge of a great variety of methods is necessary for this work.

The sixth unofficial preparation is the assay of tobacco for the nicotine content. This gives the student practice with the determination of a volatile alkaloid.

The seventh sample is a headache powder which is assayed for the per cent of caffeine and acetanilid. This method was worked out by the American Association of Official Agricultural Chemists and gives the student considerable experience in technic of separation.

The Tincture of *Nux Vomica* and the Tincture of cinchona are each assayed for the alcohol content, by determining the specific gravity of the distilled sample and also by determining the index of refraction of the distillate with the immersion refractometer.

A good deal of attention is paid to reporting all samples, especially pills and tablets, where there are many things to consider besides the assay. For example, the report on compressed tablets includes the general appearance, fracture, surfaces, uniformity of thickness and diameter, measured with the screw micrometer, time of disintegration and weight. The report on coated pills and tablets includes uniformity of shape, kind of coating, polish, coating disintegration time and uniformity of weight.

The Season's Greetings with wishes for a Merry Christmas and a more prosperous New Year.