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SOLVENTS FOR USE WITH THE MUNROE CRUCIBLE.

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Although when in using the Munroe crucible a succession of determinations of the same analytical radical in similar substances and by a uniform method of analysis are to be made, the analytical product may be allowed to accumulate in the crucible, yet if there be any variation in the analytical material or process the "residue on ignition" must be removed from the felt between each determination. In some instances a part at least may be shaken or blown out, but the felt can be completely cleansed only by the use of solvents, and this method of procedure is an essential feature of the use of the Munroe crucible in such determinations. The selection of solvents involves several considerations such as the effect of the solvent, either alone or in the presence of air or the solvent product, upon the platinum, and its capacity to easily dissolve the "residue on ignition" and to form with it a solution which is readily and completely removed from the felt. While the solubilities and properties of platinum in platinum ware have been widely studied, and precautions to be taken in its use are to be found in the literature (*vide* Prescott and Johnson, *Qual. Chem. Analysis*, **1901**, p. 95), yet the peculiar properties of platinum in a finely divided state, as it exists in platinum felt, have not been studied further than as to its catalytic action. In

the application of the various solvents for removing precipitates from the felt in the Munroe crucible, certain solubilities of the felt were observed which are not exhibited by solid platinum. The finely divided state and catalytic action of platinum in the felt suggest causes for its modified properties.

Of the solvents used to remove precipitates from the felt, hot concentrated sulphuric acid and hot hydrochloric acid, if used alone, cause a decided loss of the reduced platinum, the former by rendering the platinum colloidal, in which condition it is readily carried through by the wash-water (Phelps), and the latter by taking up the platinum as chloride (presumably by reason of the oxidation of the hydrochloric acid by the dissolved oxygen in the presence of reduced platinum which acts as a catalyzer). However, both these acids, and even fuming sulphuric acid, as well as a mixture of concentrated or fuming sulphuric acid with concentrated hydrochloric acid, may be used freely in the crucible without causing the slightest weighable loss of platinum, provided the following additions are made to the several solvents, as indicated: Ammonium salts, alkali or ammonium nitrates, or nitric acid, to concentrated or fuming sulphuric acid; ammonium salts, other than the nitrate, to the mixtures of concentrated or fuming sulphuric acid with hydrochloric acid, the practice being to add solid ammonium chloride to the sulphuric acid, thereby providing both the hydrochloric acid of the mixture and the required ammonium salt; and ammonium chloride, or oxalic acid or like reducing agent, to hydrochloric acid.

With a view to the general application of the Munroe crucible in gravimetric analysis, suitable solvents will now be given for the various precipitates, in condition for weighing. Many of these solvents have long been known, but are given here because they are suitable for use with platinum felt, while the other solvents were found in the course of investigation. In the following list appear the various solvents used with reference numbers by means of which the proper solvents may be designated for the precipitates, an alphabetical list of which follows next after the list of solvents:

Solvents.

(Salts are applied in aqueous solution unless otherwise specified.)

- | | |
|--|--|
| 1. Water. | 8. Acetic acid. |
| 2. Alcohol. | 9. Oxalic acid. |
| 3. Carbon disulphide. | 10. Hydrochloric acid with ammonium chloride or oxalic acid. |
| 4. Sulphuric acid with nitric acid or nitrates. | 11. Hydrofluoric acid. |
| 5. Sulphuric acid, concentrated or fuming, with ammonium chloride. | 12. Potassium hydroxide. |
| 6. Nitric acid. | 13. Sodium hydroxide. |
| 7. Carbon dioxide in aqueous solution. | 14. Ammonium hydroxide. |
| | 15. Potassium sulphide. |

- | | |
|----------------------------------|-----------------------------|
| 16. Potassium sulphide, yellow. | 31. Potassium chlorate. |
| 17. Sodium sulphide. | 32. Ammonium carbonate. |
| 18. Sodium sulphide, yellow. | 33. Potassium chloride. |
| 19. Ammonium sulphide. | 34. Potassium iodide. |
| 20. Ammonium sulphide, yellow. | 35. Sodium chloride. |
| 21. Potassium bisulphite. | 36. Ammonium chloride. |
| 22. Sodium thiosulphate. | 37. Ammonium fluoride, dry. |
| 23. Ammonium sulphate. | 38. Calcium chloride. |
| 24. Ammonium nitrate. | 39. Magnesium chloride. |
| 25. Sodium hydrogen phosphite. | 40. Potassium cyanide. |
| 26. Ammonium oxalate. | 41. Ferrous sulphate. |
| 27. Ammonium acetate, alkaline. | 42. Silver nitrate. |
| 28. Ammonium tartrate, alkaline. | 43. Lead acetate. |
| 29. Potassium carbonate. | 44. Mercuric nitrate. |
| 30. Sodium carbonate. | 45. Ferric acetate. |

Solvents for Precipitates in Condition for Weighing.

(Numbers indicate correspondingly numbered solvents in the preceding list. Hyphens indicate successive treatments; commas indicate alternative treatments. Abbreviations: h = hot, c = concentrated, d = dilute.)

Precipitates.	Solvents.
Aluminum oxide	12ch-1-10, 13ch-1-10.
Ammonium arsenomolybdate.	14.
Ammonium chloride.	1h
Ammonium magnesium arsenate.	6, 10
Ammonium phosphomolybdate.	1h, 12, 13, 25, 26, 29, 30, 36
Ammonium chlorplatinat.	1h
Antimony.	6-1-10
Antimony pentasulphide.	14h
Antimony tetroxide.	10ch
Antimony trisulphide.	10c, 12d, 15, 19
Arsenious sulphide.	12, 13, 15, 17, 21, 29, 30
Barium carbonate.	7, 24, 36
Barium carbonate, ignited.	1h, 6d
Barium chromate.	6, 10
Barium silicofluoride.	36
Barium sulphate.	4h, 5h
Bismuth.	6d
Bismuth carbonate (basic).	6, 10
Bismuth chromate (basic).	6
Bismuth nitrate (basic).	6
Bismuth oxide.	10
Bismuth oxychloride.	10c
Bismuth sulphide.	6ch-1-3
Cadmium carbonate.	6d, 14
Cadmium oxide.	4, 5, 6, 10, 14
Cadmium sulphide.	4dh, 6, 10
Calcium carbonate.	6d, 10d
Calcium carbonate, ignited.	1h, 6d, 10d
Calcium fluoride.	4-1, 10c
Calcium oxalate.	6, 22

Precipitates.	Solvents.
Calcium sulphate.....	23dh
Chromium oxide.....	6ch + 31dry + 42 or 43, 31dry h + 1, 42dry h + 6
Cobalt.....	6
Cobalt hydroxide.....	23, 24, 36
Cobaltous sulphate.....	1h
Cobaltous sulphide.....	4c, 5, 8c, 10c
Cupric hydroxide.....	6, 10, 14
Cupric sulphide.....	6h, 40
Cuprous oxide.....	4
Cuprous sulphide.....	6
Cuprous sulphocyanate.....	4
Gold.....	4c
Gold sulphides.....	16, 40
Iron acetate (basic).....	10
Iron arsenate.....	6, 10
Iron formate (basic).....	10
Iron hydroxide.....	6d, 10d
Iron oxide.....	10c
Iron phosphate.....	10, 45
Iron succinate (basic).....	4, 5, 6, 10
Iron sulphide.....	4, 10
Lead arsenate.....	6
Lead carbonate.....	6d
Lead chloride.....	1h
Lead chromate.....	6, 12
Lead oxalate.....	6d
Lead oxide.....	6d
Lead phosphate.....	6
Lead sulphate.....	24, 27, 28, 10ch, 6ch, 22, 12h, 13h, 14h
Lead sulphide.....	6c, 10c
Magnesium oxide.....	4d, 6d, 10d
Magnesium phosphate.....	6, 10
Magnesium pyroarsenate.....	6, 10
Magnesium pyrophosphate.....	6, 8, 10, 6ch, 10ch
Magnesium sulphate.....	1h
Manganese dioxide.....	5h + 9
Manganese sesquioxide.....	4 + 9, 4 + 41
Manganese sulphide.....	4d, 6d, 10d
Manganous pyrophosphate.....	4ch, 5h, 6ch, 10ch.
Manganous sulphate.....	1
Mercuric oxide.....	6, 10
Mercuric sulphide.....	15 + 12
Mercurous chloride.....	6ch-1
Mercurous chromate.....	6ch
Mercurous phosphate.....	6
Metastannic acid.....	10, 13
Nickel.....	6c
Nickel oxide.....	10
Nickelous hydroxide.....	10d, 14, 23, 24, 36
Nickelous subsulphide.....	6
Nickelous sulphate.....	1h

Precipitates.	Solvents.
Nickelous sulphide.....	6
Palladium iodide.....	14
Platinum sulphide.....	16, 18
Potassium chloride.....	1h
Potassium cobaltic nitrite.....	1h
Potassium fluoborate.....	1, 2d
Potassium chlorplatinate.....	1h, 12h
Potassium sulphate.....	1h
Silica.....	11, 12h, 13h, 29h, 30h, 37-ignition
Silver.....	6d
Silver chloride.....	14h
Silver cyanide.....	14, 40
Silver iodide.....	34c, 22, 40, 44ch
Silver phosphate.....	6, 14
Silver sulphide.....	6ch-1-3
Sodium carbonate.....	1h
Sodium chloride.....	1h
Sodium chlorplatinate.....	1h, 2
Sodium sulphate.....	1h
Stannic acid.....	10-1
Stannic oxide.....	36dry-ignition, 10-1
Stannic phosphate.....	12
Stannic sulphide (hydrous).....	10ch, 12, 15, 17
Stannous sulphide (hydrous).....	10c
Strontium carbonate.....	7, 24, 36
Strontium sulphate.....	33c, 35c, 38c, 39c
Uranyl pyroarsenate.....	6
Uranyl pyrophosphate.....	4, 6, 10
Zinc oxide.....	rodh
Zinc sulphide.....	4, 6, 10

A Munroe crucible may be readily prepared by igniting a carefully dried layer of ammonium chlorplatinate deposited over the bottom of a perforated platinum crucible in the form of an alcoholic sludge which is poured into the crucible while the latter is pressed upon an absorbent support to remove the excess of alcohol. The salt should be shaped during ignition, and lightly burnished thereafter, with a glass rod of suitable shape. If a felt retains cold, freshly precipitated calcium oxalate, it may be considered free from cracks and sufficiently fine to hold the finest precipitates.

In case imperfections develop in the felt, by reason of careless or improper use, it may be readily restored to prime condition by saturating it with hydrochlorplatinic acid solution, allowing a moderately strong aqueous solution of ammonium chloride to diffuse upwards throughout the felt by lowering the crucible slowly into a beaker containing the ammonium chloride solution, washing with alcohol, drying, and igniting.