

A COMPILATION AND CLASSIFICATION OF IMMERSION MEDIA OF HIGH INDEX OF REFRACTION*

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ABSTRACT

The composition of more than 100 media whose indices of refraction are stated to be equal to or greater than that of methylene iodide, $n=1.74$, are classified in four groups: (1) pure liquids, (2) liquid solutions, (3) pure solids, and (4) solids (2 or more components). Many liquid immersion media are not adequate for routine use in the mineralogical laboratory because of their reactivity with the immersed phase, their instability and toxicity. Five new liquids whose indices of refraction are 1.99–2.07 have been discovered. They are arsenic tribromide solutions of various mixtures containing sulfur, selenium, arsenic disulfide, arsenic trisulfide, and mercuric sulfide.

INTRODUCTION

The use of immersion media in the identification of crystalline material is well established, and satisfactory immersion liquids have long been available for indices of refraction below $n=1.74$. The unsatisfactory nature of available high index liquids prompted a study of the possibility of developing new and better liquids, and some have already been reported (Meyrowitz and Larsen, 1951; Meyrowitz, 1952). Five new liquids (II B 6, 7, 8, 9, 10), have since been discovered. Their preparation and properties will be described in future publications.

In the course of this work, the literature on proposed media of high index of refraction has been reviewed. The present paper is a compilation of all such media, classified into four categories:

- I. Pure liquids;
- II. Liquid solutions, further subdivided by solvents;
- III. Pure solids;
- IV. Solids (2 or more components).

These media are arranged alphabetically and the corresponding indices of refraction are those found in the original references. In this paper is reported the highest index for that composition. Where a complete series of media has been made, the range of indices is given. The chemical formulas of the more uncommon compounds are given. A question mark (?) after the name of a compound indicates either an ambiguity in the naming of a compound or that the name is obsolete and/or the compound cannot be identified.

Most of the solid media have been proposed by biologists and botanists as mounting media for diatoms and other biological specimens.

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Desirable properties of high index media for routine use in the mineralogical laboratory are:

(1) They should be liquids. (2) They should be stable (the maximum change in index of refraction should not be more than a few units in the third decimal place in one year). (3) They should not constitute a health or safety hazard. (4) They should be inert to all minerals.

Although embedding media are neither as convenient nor as accurate as liquid immersion media (Larsen and Berman, 1934, p. 13), their use is necessary if indices above 2.1 are to be measured.

HIGH INDEX MEDIA

I. *Pure liquids*

- A. Methylene iodide, CH_2I_2 , $n=1.74$ (Brauns, 1886, p. 77).
- B. Phenyl-di-iodoarsine, $\text{C}_6\text{H}_5\text{AsI}_2$, $n=1.85$ (Gryszkiewicz-Trochimowski and Sikorski, 1928, p. 411-412).
- C. Selenium monobromide, Se_2Br_2 , $n=2.1$ (Borgström, 1929, p. 61); $n=1.96$ (Anderson and Payne, 1934, p. 67).

Phenylsulfide, $(\text{C}_6\text{H}_5)_2\text{S}$, and dimethylmercury, $(\text{CH}_3)_2\text{Hg}$, have not been included in the list of media. The index of phenylsulfide was reported to be 1.95 (Pulfrich, 1890, p. 65; Zirkel, 1893, p. 40; Behrens, 1898, p. 46), although Abbe (1879*A*, p. 41, 1879*B*, p. 649) listed it as 1.62. However, as early as 1898, Schroeder van der Kolk (1898, p. 13) warned that it did not always have an index of 1.95, and later the index was corrected to 1.63 by Schroeder van der Kolk and Beekman (1906, p. 10). This was confirmed by Himmelbauer (1909) and by Vogel and Cowan (1943, p. 22). Johannsen (1918, p. 260) has suggested that this confusion is a result of a typographical error, an inverted "6" in the original reference. It is possible that the compound or mixture reported to have an index of 1.95 is something other than phenylsulfide. Bolland (1910, p. 389) has listed in his series of immersion liquids used in the identification of crystalline chemical compounds, a substance having a refractive index of 1.95 which he calls "phenylensulfid." A number of compounds might be confused with phenylsulfide, $(\text{C}_6\text{H}_5)_2\text{S}$, (diphenylsulfide). They are phenyldisulfide, $(\text{C}_6\text{H}_5)_2\text{S}_2$, (diphenyldisulfide); diphenylene disulfide, $(\text{C}_6\text{H}_4)_2\text{S}_2$, (thianthrene); diphenylene sulfide, $(\text{C}_6\text{H}_4)_2\text{S}$, (dibenzthiophene); and diphenylene 2:2 disulfide, $(\text{C}_6\text{H}_4)_2\text{S}_2$. However, all except phenylsulfide are solids.

Dimethylmercury (quecksilbermethyl) was listed as having an index of 1.93 (Pulfrich, 1890, p. 65; Zirkel, 1893, p. 40; Behrens, 1898, p. 47). This index is incorrect and should have been 1.53 (Marvel and Calvery, 1923, p. 823). Pulfrich (1890), Zirkel (1893) and Behrens (1898) have all listed the incorrect indices of both phenylsulfide and dimethylmercury;

all probably used the same, but faulty, original reference or each used the earlier work and accepted it as being reliable.

II. *Liquid solutions*

A. Aniline, $C_6H_5NH_2$, and quinoline, C_9H_7N , combined as a solvent.

1. Mercuric iodide (Pulfrich, 1890, p. 66; Zirkel, 1893, p. 40; Schroeder van der Kolk, 1898, p. 12); $n=1.8$ (Wright, 1911, p. 98).

Aniline darkens on exposure to air and light; it is flammable and poisonous. Quinoline darkens on storage and has a penetrating odor.

B. Arsenic tribromide as a solvent.

1. Arsenic disulfide, As_2S_2 (Smith, 1885, p. 162); $n=1.88$ (Borgström, 1929, p. 60).
2. Arsenic disulfide and arsenic triiodide; $n=1.96$ (Borgström, 1929, p. 61).
3. Arsenic disulfide and selenium (Borgström, 1933, p. 29); $n=2.11$ (Meyrowitz and Larsen, 1951, p. 749).
4. Arsenic disulfide and sulfur (Smith, 1886A, p. 76; Meates, 1886B, p. 357; Kolbe, 1927, p. 205); $n=2.00$ (Meyrowitz and Larsen, 1951, p. 748).
5. Arsenic trisulfide and selenium, $n=1.97$, red but is transparent to red, yellow, and green light (Borgström, 1933, p. 29); $n=2.04$ (Meyrowitz, unpublished data).
6. Arsenic trisulfide and sulfur, $n=2.07$ (Meyrowitz, unpublished data).
7. Sulfur, mercuric sulfide, and arsenic disulfide, $n=1.99$ (Meyrowitz, unpublished data).
8. Sulfur, mercuric sulfide, and arsenic trisulfide, $n=2.01$ (Meyrowitz, unpublished data).
9. Sulfur, selenium, and arsenic disulfide, $n=2.02$ (Meyrowitz, unpublished data).
10. Sulfur, selenium, and arsenic trisulfide, $n=2.00$ (Meyrowitz, unpublished data).

Solutions containing arsenic tribromide attack the lead-glass prisms of refractometers (Alexander, 1934, p. 181), will corrode metal, and are by their nature, poisonous and reactive toward some minerals (Meyrowitz and Larsen, 1951, p. 747). Although arsenic tribromide liquids hydrolyze in contact with water vapor, they are relatively stable (Meyrowitz and Larsen, 1951, p. 747; Meyrowitz, 1952, p. 853–855).

C. α -Bromonaphthalene, $C_{10}H_7Br$, as a solvent.

1. Arsenic tribromide, $n=1.72$ (Pulfrich, 1890, p. 65; Behrens, 1898, p. 46).
2. Arsenic tribromide and sulfur, $n=1.66$ – 1.81 (Meyrowitz, 1952, p. 854).
3. Arsenic tribromide, sulfur, and arsenic disulfide, $n=1.66$ – 2.02 (Meyrowitz, 1952, p. 855–856).
4. Phenyl-di-iodoarsine, $n=1.85$ (Bosazza, 1940, p. 300).

D. Cacodyl selenide, $[(CH_3)_2As]_2Se$, as a solvent.

1. Selenium, $n=1.83$ (Anderson, 1934, p. 221).

Cacodyl selenide has a repugnant and extremely penetrating odor.

E. Carbon disulfide, CS_2 , as a solvent.

1. Phosphorus, $n=2.01$ (Gladstone and Dale, 1859, p. 32; Stephenson, 1873, p. 2; Stephenson, 1880, p. 565; Retgers, 1893, p. 134).
2. Sulfur, $n=1.75$ (Stephenson, 1880, p. 565; Behrens, 1898, p. 50).
3. Sulfur and phosphorus, $n=2.07$ (Borgström, 1929, p. 62).

Carbon disulfide is highly volatile, very flammable and poisonous.

Solutions containing white phosphorus must be kept in the dark as light causes the conversion of the white phosphorus to the red form, which is insoluble (Madan, 1898*A*, p. 102; 1898*B*, p. 280; West, 1936, p. 247). Because of its poisonous nature and the ease with which finely divided white phosphorus undergoes spontaneous combustion it must be handled with care (Borgström, 1929, p. 62; Milton, 1948, p. 512–513; Burri, 1950, p. 225). The elements, sulfur and phosphorus, dissolved in carbon disulfide will combine chemically, frequently with explosive violence, when the mixture is heated (Thorne and Roberts, 1949, p. 751). Mixtures of sulfur and phosphorus in methylene iodide should be stored in the cold. The free iodine liberated as a result of the slow decomposition of methylene iodide probably increases the danger of explosion, for it has been reported that iodine in a mixture of sulfur and phosphorus in carbon disulfide lowers the temperature at which the elements will combine (Thorne and Roberts, 1949, p. 751). These liquids (II *J* 14.) should be kept under water (Borgström, 1929, p. 62; West, 1936, p. 247). Although hydrogen sulfide gas is evolved when these liquids are stored under water (West, 1936, p. 248), it has been shown that solutions of phosphorus and sulfur in methylene iodide are relatively stable on long storage (Brunn and Barth, 1947).

F. Diethylselenium, $(C_2H_5)_2Se$, as a solvent.

1. Sulfur and selenium, $n=1.90$ (Marpmann, 1898, p. 8).

Diethylselenium has an obnoxious odor.

G. Dimethylmercury, $(CH_3)_2Hg$, as a solvent.

1. Phosphorus, $n=1.93$ (Retgers, 1893, p. 134).

Dimethylmercury is volatile, flammable, and a deadly poison.

H. Glycerol, $C_3H_8O_3$, as a solvent.

1. Potassium iodide and mercuric iodide, $n=1.78-1.80$ (Amann, 1896, p. 21).

I. α -Iodonaphthalene as a solvent.

1. Iodoform, $n=1.81$ (Bryant, 1943, p. 97, 101).

J. Methylene iodide as a solvent.

1. Antimony tribromide, $n=1.83$ (Zirkel, 1893, p. 48; Borgström, 1933, p. 28).
2. Antimony tribromide, arsenic disulfide, sulfur, and antimony trisulfide, $n=1.96$ (Wright, 1911, p. 98).
3. Antimony tribromide and stannic iodide, $n=1.86$ (Borgström, 1933, p. 28).
4. Antimony triiodide, $n=1.76$ (Merwin, 1913, p. 36).
5. Antimony triiodide and stannic iodide, $n=1.83$ (Merwin, 1913, p. 36).
6. Arsenic tribromide, $n=1.78$ (Borgström, 1933, p. 58).
7. Arsenic tribromide and arsenic disulfide, $n=1.90$ (Borgström, 1929, p. 60–61).
8. Arsenic tribromide, arsenic disulfide and arsenic triiodide, unstable (Borgström, 1929, p. 61).
9. Arsenic tribromide and sulfur, $n=1.74-1.81$ (Meyrowitz and Larsen, 1951, p. 747–748).
10. Arsenic trisulfide, $n=1.74-2.28$ (Merwin, 1913, p. 36–37; Weinzierl, 1919/1920, p. 182; Bryant, 1932, p. 3761).

Kolbe (1927, p. 205) reports Weinzierl as having proposed a medium of arsenic disulfide (realgar) dissolved in methylene iodide which does not have the index (2.2–2.4) given by Weinzierl (1919/1920, p. 182). Kolbe says this solution has an index of 1.73 (methylene iodide). Kolbe is in error, because Weinzierl's article describes a solution, not of arsenic disulfide, but of arsenic trisulfide in methylene iodide.

11. Phenyl-di-iodoarsine, $n=1.74-1.85$ (Bryant, 1943, p. 97).
12. Phosphorus, $n=1.95$ (Madan, 1898A, p. 101; Gavelin, 1916, p. 293).
13. Phosphorus and iodine, $n=1.95$ (Zirkel, 1893, p. 40).
14. Phosphorus and sulfur, $n=1.78-2.06$ (West, 1936, p. 246).
15. Selenium monobromide, $n=1.97$ (Anderson and Payne, 1934, p. 67).
16. Selenium monobromide and sulfur, $n=1.95$ (De Ment, 1949, p. 102).
17. Selenium monobromide, sulfur, and tetraiodoethylene, C_2I_4 , $n>1.90$ (De Ment, 1949, p. 102).
18. Selenium monobromide, sulfur, tetraiodoethylene, antimony triiodide, and/or arsenic sulfide (?), $n=1.96$ (De Ment, 1949, p. 102).
19. Stannic iodide, $n=1.78$ (Merwin, 1913, p. 36).
20. Stannic iodide, arsenic triiodide, and antimony triiodide (Davies and Hartshorne, 1934, p. 1831).
21. Stannic iodide, arsenic triiodide, antimony triiodide, and iodoform (Larsen, Witt, and Poe, 1948, p. 5).
22. Stannic iodide, arsenic triiodide, antimony triiodide, and sulfur, $n=1.85$ (Merwin, 1913, p. 36).
23. Stannic iodide, arsenic triiodide, antimony triiodide, sulfur, and iodoform, $n=1.87$ (Merwin, 1913, p. 36).
24. Stannic iodide, arsenic triiodide, iodoform, sulfur, and arsenic sulfide (?), $n=1.85$ (McCormick, 1936, p. 9).

Solutions of iodides in methylene iodide are unstable and relatively dark in color (Borgström, 1929, p. 58; Jelley, 1934, p. 242; Larsen, Witt, and Poe, 1948, p. 5; Burri, 1950, p. 225).

25. Stannic iodide, arsenic triiodide, and iodoform, $n=1.84$ (Merwin, 1913, p. 36).
26. Stannic iodide and sulfur, $n=1.82$ (Merwin, 1913, p. 36).
27. Sulfur, $n=1.78$, (Bertrand, 1888 p. 31; Madan, 1898A, p. 101; Wright, 1911, p. 98).
28. Sulfur and arsenic, $n=1.85-2.01$ (Bryant, 1938, p. 1397).

Solutions of sulfur and arsenic in methylene iodide are unstable (H. E. Merwin, personal communication) as are solutions of arsenic trisulfide in methylene iodide (Merwin, 1913, p. 37).

29. Sulfur and arsenic trisulfide, $n=1.9$ (Plato, 1942, p. 238).
30. Sulfur and iodine, $n>1.85$ (Bertrand, 1888); $n=1.82$ (Johannsen, 1918, p. 252).
31. Sulfur and iodoform, $n=1.81$ (McCormick, 1936, p. 9).
32. Sulfur and tetraiodoethylene, $n=1.81$ (Anderson, 1933, p. 203).
33. Sulfur, tetraiodoethylene, antimony triiodide, and arsenic sulfide (?), $n=1.96$ (De Ment, 1949, p. 102).
34. Sulfur, tetraiodoethylene, and stannic iodide, $n=1.83$, tends to attack the glass parts of refractometers (Anderson, 1933, p. 203).

The frequent use of methylene iodide as a solvent in the preparation of high index immersion liquids is due primarily to its high index of refraction and its ability to dissolve relatively large amounts of solids. The volatility of methylene iodide is less than that of carbon disulfide but more than that of α -bromonaphthalene. It is inert to most minerals but has the disadvantage of decomposing slowly on exposure to light, air, and water, becoming dark because of the liberation of free iodine. Copper shot or wire placed in the liquid has been used to preserve its light color. As its usefulness as a heavy liquid for mineral separation and as a high index immersion liquid depends on its purity, the commercially produced reagent, although now available with reasonable purity, should be tested to verify that its index of refraction ($n_D^{25^\circ \text{C}}$) is 1.737.

K. Phenyldi-iodoarsine as a solvent.

1. Arsenic sulfides, $n=1.93$ (Borgström, 1951, p. 240).
2. Arsenic trisulfide and selenium, $n=1.95$ (Borgström, 1951, p. 240).

Borgström (1951, p. 239–240) has urged the use of phenyldi-iodoarsine as a solvent in mixtures in order to obtain high index liquids. It has a blistering action on the skin (Anderson and Payne, 1934, p. 66) and is known to attack the soft glass of refractometers (Anderson and Payne, 1936, p. 168).

L. Phosphorus as a solvent.

1. Selenium, $n=2.2$ – 2.5 (Retgers, 1894, p. 424); $n=2.17$ (Borgström, 1929, p. 62).
2. Sulfur, $n=2.2$ – 2.5 (Retgers, 1894, p. 424); $n=2.07$ (Borgström, 1929, p. 62).
3. Sulfur and selenium (Borgström, 1929, p. 62).

Solutions of selenium in phosphorus cannot be stored under water because they decompose, as is shown by the appearance of red flakes of selenium in the protective water layer (Borgström, 1929, p. 63).

M. Selenium monobromide as a solvent.

1. Selenium, $n>2.02$ (Borgström, 1929, p. 61; Anderson and Payne, 1934, p. 67).
2. Selenium, methylene iodide, sulfur, tetraiodoethylene, $n>1.90$ (Anderson and Payne, 1934, p. 67).

Selenium monobromide is unstable, decomposing with the separation of selenium, and it is almost opaque except in thin film (Borgström, 1929, p. 61). It is unpleasant to handle (Anderson and Payne, 1940, p. 580).

N. Water as a solvent.

1. Barium mercuric iodide, $n=1.79$ (Rohrbach, 1883, p. 172; Schroeder van der Kolk, 1898, p. 12).
2. Mercuric nitrate-mercuric iodide, $n=1.80$ (Zirkel, 1893, p. 40).
3. Potassium mercuric iodide, $n=1.73$ (Goldschmidt, 1881, p. 234; Stephenson, 1882, p. 167; Merwin, 1911, p. 427).

III. *Pure solids*

- A. Antimony pentasulfide (Morris, 1885*B*, p. 132).
 B. Antimony triiodide (Morris, 1885*B*, p. 130).
 C. Antimony trisulfide (Morris, 1885*B*, p. 132).
 D. Arsenic (Hanna and Grant, 1939, p. 175).
 E. Arsenic disulfide, $n=2.45$, (Gifford, 1892, p. 174; Marpmann, 1897, p. 338; Hanna and Grant, 1939, p. 175).
 F. Arsenic selenide, As_2Se_3 (Merwin in Larsen and Berman, 1934, p. 17).
 G. Arsenic tribromide, $n=1.78$ (Abbe, 1879*A*, p. 42, 1879*B*, p. 650; Rohrbach, 1883, p. 172; Pulfrich, 1890, p. 65; Zirkel, 1893, p. 40; Madan, 1898*B*, p. 273; Borgström, 1929, p. 58–59).
 H. Arsenic triiodide (Morris, 1885*B*, p. 129).
 I. Chloro-chromic acid, (?), CrO_2Cl_2 (Morris, 1885*B*, p. 128).
 J. Chromic acid, CrO_3 (Morris, 1885*B*, p. 127).
 K. Cyanogen iodide, CNI (Morris, 1885*B*, p. 130).
 L. Lead chromate (Gladstone and Dale, 1859, p. 30).
 M. Mercuric iodide (Morris, 1885*B*, p. 130).
 N. Phosphorus, $n=2.1$ (Gladstone and Dale, 1859, p. 30–31; Stephenson, 1873, p. 2; Damien, 1881, p. 269, 271; Retgers, 1893, p. 131).
 O. Selenium (Hanna and Grant, 1939, p. 175).
 P. Selenium tetrachloride (Morris, 1885*B*, p. 131).
 Q. Silver bromide (Morris, 1885*B*, p. 130), $n=2.25$ (De Ment, 1949, p. 102).
 R. Silver chloride, $n=2.07$ (De Ment, 1949, p. 102).
 S. Silver iodide (Morris, 1885*B*, p. 130).
 T. Sulfur, $n=2.1$ (Stephenson, 1880, p. 565; Morris, 1885*B*, p. 125; Marpmann, 1897, p. 338; Schroeder van der Kolk, 1899, p. 627).
 U. Tellurium (Morris, 1885*A*; p. 76; Hanna and Grant, 1939, p. 175).
 V. Tellurium chloride (Morris, 1885*B*, p. 130).
 W. Thallium monobromide, $n=2.42$ (De Ment, 1949, p. 102).
 X. Thallium monochloride (Morris, 1885*B*, p. 131), $n=2.25$ (De Ment, 1949, p. 102).
 Y. Thallium monoiodide (Morris, 1885*B*, p. 131).

IV. *Solids (2 or more components)*

- A. Antimony tribromide, glycerol and
 1. Boracic acid, H_3BO_3 , $n > 1.7$ (Smith, 1886*B*, p. 3; 1886*C*, p. 13–14).
 2. Arsenious acid, n =approximately 2 (Smith, 1886*A*, p. 76; Strasburger and Koernicke, 1923, p. 441).
 B. Arsenic tribromide and arsenic disulfide, $n=2.4$ (Smith, 1885, p. 162, 1886*A*, p. 76; Meates, 1886*B* p. 357; Van Heurck, 1886–1887, p. 22–23).
 C. Antimony trichloride and arsenic (Morris, 1885*B*, p. 129).
 D. Arsenic disulfide and
 1. Mercuric iodide (Morris, 1885*B*, p. 132).
 2. Sodium tetraborate, $Na_2B_4O_7$ (Marpmann, 1897, p. 338).
 E. Lead chromate and chromic acid (Morris, 1885*B*, p. 127).
 F. Piperine, $C_{17}H_{19}NO_3$ and
 1. Antimony tribromide, n =approx. 1.70 (Van Heurck, 1907, p. 60).
 2. Antimony triiodide and arsenic triiodide, $n=1.68$ –2.10 (Merwin, 1913, p. 37; Billiet, 1925, p. 113–114; Fisk, 1930, p. 263–266).
 3. Mercuric iodide (Morris, 1885*B*, p. 126).

G. Selenium and

1. Arsenic (Marpmann, 1898, p. 6-7).
2. Arsenic disulfide, $n > 2.5$ (Jones, 1914, p. 25).
3. Arsenic triselenide, $n = 2.72-3.17$ (Merwin in Larsen and Berman, 1934, p. 17).

The melt containing selenium and arsenic trisulfide described by Burri (1950, p. 226) is incorrect and will be corrected in a future edition of his book (Burri, personal communication). It should be "selenium and arsenic selenide."

H. Sulfur and

1. Arsenic (Meates, 1886A; p. 171; Marpmann, 1898, p. 7).
2. Arsenic disulfide (Morris, 1885B, p. 132; Gifford, 1892, p. 174).
3. Arsenic disulfide and mercuric iodide (Morris, 1886, p. 100).
4. Arsenic trisulfide, $n = 2.1-2.6$ (Merwin, 1913, p. 37).
5. Arsenious acid and bromine (Nelson, 1892, p. 123).
6. Selenium (Morris, 1885B, p. 125; Marpmann, 1898, p. 7); $n = 1.99-2.92$ (Merwin and Larsen, 1912, p. 42; Billiet, 1925, p. 113-115).

I. Synthetic resins

1. Aniline, $C_6H_5NH_2$, formaldehyde, CH_2O , and sulfur, $n = \text{approx. } 1.80$ (Hanna, 1930, p. 424-425).
2. Hyrax (derivative of naphthalene, $C_{10}H_8$), $n = 1.82$ (Hanna, 1930, p. 425).
3. Naphthalene, formaldehyde, acetic acid and sulfuric acid, $n = 1.76-1.80$ (Fleming, 1943, p. 35-36).

J. Tellurium and arsenic selenide (Merwin in Larsen and Berman, 1934, p. 17).

K. Thallium monobromide and thallium monoiodide, $n = 2.4-2.8$ (Barth, 1929, p. 358-361).

L. Thallium monochloride and

1. Stannic chloride (Morris, 1885B, p. 131).
2. Valerianate (?) of quinine (Morris, 1885B, p. 131).

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