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DIELECTRIC DILUENT

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This invention relates to insulating fluids, such as those particularly adapted for use in electrical equipment. In electrical apparatus such as transformers, switches, electric bushings, cables, fuses, and similar equipment, it is usual to provide an insulating medium which is ordinarily termed the "dielectric." This invention is concerned with certain novel and improved dielectric liquids which possess superior electrical properties, are non-flammable and do not evolve inflammable gases when subjected to decomposition.

More particularly, this application is concerned with certain diluents which have been found especially useful for use in diluting and lowering the freeze point of various dielectric compositions. These diluent liquids are especially useful for diluting compositions prepared as described in our co-pending applications Ser. No. 53,099 filed December 5, 1935, and Ser. No. 68,919, filed March 14, 1936, to which this applications bears the relationship of continuation in part. However, it is not restricted in use to the dielectric compositions described in these co-pending applications but is of broad general utility wherever it is desired to add a diluent liquid to a dielectric composition for purposes of lowering the freeze point, viscosity, or other properties of the dielectric composition and wherein it is desired that this diluent impart non-inflammability or non-flammability to the resulting composition.

A dielectric or insulating liquid in some pieces of electrical equipment such as in transformers, serves the dual function of acting as an insulating medium and also taking up and conveying to cooling surfaces heat generated within the apparatus. In various installations dielectric liquids are subjected to considerable heat and sometimes to relatively high temperatures. This is especially true where failures occur in the electrical equipment resulting in short circuits with the consequent development of very high temperatures. It is therefore apparent that the diluent liquid which is added to dielectric compositions must be such that the composition is either rendered non-inflammable or at least the fire hazard not increased by the addition of the diluent. The dielectric diluents with which this application is concerned are substantially non-inflammable and when added in diluent amounts to certain dielectric compositions actually improve the characteristics of the dielectric insofar as fire point and flash point is concerned.

The important function usually served by the addition of a diluent liquid to a dielectric composition is the lowering of the freeze point. The freeze point of a dielectric is usually defined as that temperature at which crystals deposit upon prolonged cooling. Obviously, in various installations, electrical equipment containing dielectric liquids are subjected to relatively low temperatures, and it is essential that freezing of the dielectric shall not occur, at least not to any substantial extent. Moreover, in some installations the dielectric liquid is circulated in order to keep it relatively cool and it is frequently essential that a diluent be added to such a dielectric liquid in order to reduce the viscosity of such compositions at relatively low temperatures to such an extent that they may be economically and efficiently circulated.

The product which we propose to utilize as a dielectric diluent for dielectrics in electrical installations consists essentially of isopropyl tetrachlorobenzene, which is prepared by chlorinating isopropyl benzene (cumene) until there are present from 3.5 to 4.5 atoms of chlorine per mole of isopropylbenzene. This chlorination should be carried out under conditions wherein chlorination of the isopropyl side chain is substantially avoided. This means that the temperature of chlorination should be maintained within the temperature range 0 to 75° C. and preferably within the range 40 to 60° C. The chlorination should be carried out in the absence of light and in the presence of any suitable halogenation catalyst favoring the substitution of a halogen in the nucleus such as iron, iodine, ferric chloride, etc. We prefer to use iron, however, because of its cheapness, and because of the ease with which it may subsequently be removed from the product. Ordinarily from 1 to 5% of iron filings or iron scrap is utilized as the catalytic agent.

In preparing our improved dielectric diluents, we slowly bubble chlorine into isopropyl benzene (cumene). Hydrogen chloride is evolved during the reaction and passes off into the atmosphere. If desired, it may be recovered. The process is carried out in the absence of light and in the presence of some halogenation catalyst favoring the substitution of halogen in the nucleus, as specified above. From 3.5 to 4.5 atoms of chlorine per mole of cumene are introduced and the product may then be taken and used directly as a freeze point depressant or diluent for various dielectric compositions or, if desired, it may be split by fractional distillation and condensation into various fractions, one boiling between 260 to 310° C. being especially valuable as a diluent and freeze point depressant.

As an indication of the character of our product various properties will be given of various fractions of isopropyl chlorobenzene, containing from 3.5 to 4.5 atoms of chlorine per mole of isopropyl benzene. In each case the product has been listed as comprising three fractions, that boiling between 270 to 310° C., the fraction including lower boiling material which boils between 260 and 310° C., and the fraction includ-

ing still lower boiling material which boils between 250 and 310° C. Any one of these fractions may be utilized as freeze point depressants and diluents in accordance with our invention. In this table the melting point is that temperature at which all crystals disappear on heating.

TABLE I

No. of chlorine atoms per mole of isopropyl benzene	3.5			4.0			4.5		
Fraction	I	II	III	I	II	III	I	II	III
Boiling range of fraction...°C.	270–310	260–310	250–310	270–310	260–310	250–310	270–310	260–310	250–310
Density at 96° C.	1.33	1.32	1.32	1.36	1.35	1.35	1.44	1.43	1.43
Melting point...°C.	−18	17	23	30	29	35	57	51	50
Freeze point...°C.	Below −40	−25	−7	10	7	15	45	34	30
Flash point...°C.	180	180	175	180	180	165	Over 200	Over 200	Over 200
Fire point...°C.	None	None	223	None	None	None	None	None	None

NOTE.—The melting point is the temperature at which all crystals disappear. The freeze point is the temperature at which crystals first appear upon cooling.

While the dielectric diluents that we have described may be utilized for lowering the freeze point of any dielectric composition, we have found them especially suitable for use in connection with the compositions described in our previously referred to co-pending applications. The diluents may be added for various purposes to compositions comprising ethyl trichlorbenzene and ethyl tetrachlorbenzene; ethyl trichlorbenzene, ethyl tetrachlorbenzene and ethyl pentachlorbenzene; or ethyl tetrachlorbenzene and ethyl pentachlorbenzene; such as those described in our application Ser. No. 53,099. As described in that application the preferred chlorinated ethyl benzene composition contains from 4.0 to 4.8 atoms of chlorine per mole of ethylbenzene, and that composition containing up to 40% of our novel diluents (which are prepared by chlorinating cumene until the chlorine content is 3.5 to 4.5 atoms per mole of cumene) may be regarded as one of our preferred embodiments. When 20% of the specified diluent is incorporated with the composition of Ser. No. 53,099 containing from 4.0 to 4.8 atoms of chlorine per mole of ethylbenzene the freeze point of the resulting composition is lowered to below −40° C. Moreover, the desirable properties of these chlorinated products, so far as their relatively high flash and fire points are concerned, are not deleteriously affected by the addition of the diluent.

As an illustration of the characteristics of ethylchlorobenzenes containing from 4.0 to 4.8 atoms of chlorine per mole of ethyl benzene, with which our dielectric diluents are especially adapted for use, the following summary is given. As will be noted it is possible to separate these ethylchlorobenzenes containing 4.0 to 4.8 atoms of chlorine per mole of ethylbenzene into three fractions having boiling points within the ranges 260 to 270° C., 270 to 285° C., and 285 to 305° C. respectively.

TABLE II

Ethyl chlorobenzene dielectric compositions

Fraction	Boiling range	Density at 30° C.	Temp. at which crystals appear on prolonged cooling	Temp. at which crystals disappear on warming
	° C.		° C.	° C.
I	260–270	1.40–1.45	Below 0	Below 0
II	270–285	1.45–1.50	Below 0	10
III	285–305	1.50–1.55	20–25	30–40

As illustrative of the use of chlorinated isopropyl benzene containing from 3.5 to 4.5 atoms of chlorine per molecule of isopropyl benzene as a freeze point diluent for lowering the freeze point of ethyl tetrachlorobenzene-ethyl pentachlorobenzene mixtures the following compositions and experimentally determined freeze points may be given:

TABLE III

Freeze pt. lowering of ethyl tetra-pentachlorobenzenes by isopropyl chlorobenzenes (3.5–4.5 Cl/mol.)

Composition	Percent of ethyl tetra-chlorbenzene-penta chlorbenzene in dielectric compositions	Percent of isopropyl chlorbenzene containing substantially 3.5 atoms of Cl_2 per mole of isopropyl benzene in the dielectric composition	Percent of isopropyl chlorbenzene containing substantially 4.0 atoms of Cl_2 per mole of isopropyl benzene in the dielectric composition	Percent of chlorinated isopropyl-benzene containing 4.5 atoms of Cl_2 per mole of isopropyl benzene in the dielectric composition	Freeze point, ° C.	Melting point °C.
1	100				+12	+23
2		100			−7	+23
3			100		−8	+20
4				100	+30	+50
5	67	33			−33	+2
6	33	67			Below −60	
7	67			33	Below −40	
8	33			67	+8	+32
9	83		17		−2	+17
10	72		28		−17	+11
11	62.5		37.5		Below −50	
12	56		44		Below −50	
13	28		72		−50	
14	17		83		−28	+17

It is thus obvious that amounts of chlorinated isopropyl benzene up to about 40%, which is the percentage of our novel diluent which we prefer to add, lower the freeze point of this particular ethyl tetrachlorobenzene-ethyl pentachlorobenzene fraction to below about −50° C. As will be noted from the table, when the composition con-

tains 83% of chlorinated isopropyl benzene having a chlorine content of 4.0 atoms of chlorine per molecule of isopropyl benzene and but 17% of the ethyl tetrachlorobenzene-ethyl pentachlorobenzene mixture, the freeze point is somewhat higher, being approximately —28° C.

Our diluents are also adapted for use in depressing the freeze point of the various isopropyl chlorbenzenes disclosed in our copending application, Ser. No. 68,919. These products may contain from 3.5 to 5.0 atoms of chlorine per mole of isopropyl benzene and may have freezing points varying from —40° C. to 58° C. Some of these products are solids at room temperature and it is, of course, essential in some installations that they be rendered liquid by the use of a freeze point depressant or a diluent such as those forming the subject matter of this application.

It is to be understood that the various compositions herein disclosed may be varied somewhat in composition without departing from the scope of our invention. We do not desire that the various numerical proportions and properties of the dielectric diluents and mixtures herein disclosed be restricted except as necessitated by the scope of the appended claims.

Wherever in the appended claims conditions favoring the substitution of chlorine in the nucleus, or conditions of chlorination such that chlorination of the isopropyl group is substantially avoided, are specified these conditions are those previously set forth; i. e., the temperature of chlorination must be maintained within the temperature range 0–75° C., preferably within the range 40–60° C., and the chlorination is carried out in the absence of light and in the presence of a halogenation catalyst such as iron, iodine or ferric chloride. Wherever in the claims reference is made to isopropyl tetrachlorobenzene, the product so defined is the material resulting from the chlorination of isopropylbenzene with chlorine under the previously specified conditions such that chlorination of the isopropyl side chain is substantially avoided, until there have been absorbed from 4.0 to 4.5 atoms of chlorine per molecule of isopropylbenzene, said product being substantially completely free from the chemical compound having an atmospheric boiling point of 252 to 253° C. and known as isopropyl trichlorobenzene.

We claim:

1. An electrical insulating composition comprising ethyl trichlorobenzene, ethyl tetrachlorobenzene, and isopropyl tetrachlorobenzene.

2. An electrical insulating composition comprising ethyl trichlorobenzene, ethyl tetrachlorobenzene, and, as a diluent, nuclear chlorinated isopropyl benzene containing from 4.0 to 4.5 atoms of chlorine per mole of isopropyl benzene.

3. An electrical insulating composition comprising ethyl trichlorobenzene, ethyl tetrachlorobenzene, ethyl pentachlorobenzene and isopropyl tetrachlorobenzene.

4. An electrical insulating composition which comprises ethyl trichlorobenzene, ethyl tetrachlorobenzene, ethyl pentachlorobenzene and, as a dielectric diluent, nuclear chlorinated isopropyl benzene containing from 4.0 to 4.5 atoms of chlorine per mole of isopropyl benzene.

5. An electrical insulating composition which comprises ethyl tetrachlorobenzene, ethyl pentachlorobenzene, and isopropyl tetrachlorobenzene.

6. An electrical insulating composition comprising ethyl tetrachlorobenzene, ethylpentachlorobenzene and, as a diluent, nuclear chlorinated isopropyl benzene containing from 4.0 to 4.5 atoms of chlorine per mole of isopropyl benzene.

7. An electrical insulating composition comprising ethyl pentachlorobenzene and isopropyl tetrachlorobenzene.

8. An electrical insulating composition comprising ethyl pentachlorobenzene and, as a diluent and freeze point depressant, nuclear chlorinated isopropyl benzene containing from 4.0 to 4.5 atoms of chlorine per mole of isopropyl benzene.

9. An electrical insulating composition comprising ethyltrichlorobenzene, ethyltetrachlorobenzene and, as a freeze point depressant and diluent, nuclear chlorinated isopropylbenzene containing from 4.0 to 4.5 atoms of chlorine per mole of isopropylbenzene.

10. An electrical insulating composition which comprises nuclear chlorinated ethylbenzene containing from 3 to 5 atoms of chlorine per mole of ethylbenzene and, as a dielectric diluent and freeze point depressant, nuclear chlorinated isopropyl benzene containing from 4.0 to 4.5 atoms of chlorine per mole of isopropyl benzene.

11. An electrical insulating composition comprising nuclear chlorinated ethylbenzene containing from 3.0 to 5.0 atoms of chlorine per mole of ethylbenzene and, as a dielectric diluent therefor, isopropyl tetrachlorobenzene.

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