



# PATENTBESVÄRSRÄTTENS DOM

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## **PARTER**

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## **SAKEN**

Upphävande av patent på ytbeklädnadsmaterial i laminatform samt isoleringssystem innefattande ett dylikt ytbeklädnadsmaterial

## **ÖVERKLAGAT AVGÖRANDE**

Patent- och registreringsverkets (PRV) beslut den 19 mars 2010 angående patent nr 0401043-5, se bilaga 1

## **DOMSLUT**

Patentbesvärsrätten avslår överklagandet.

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**REDOGÖRELSE FÖR SAKEN OCH FRAMSTÄLLDA YRKANDEN**

Saint-Gobain Isover AB (Isover) beviljades den 6 december 2005 patent på "Ytbeklädnadsmaterial i laminatform samt isoleringssystem innefattande ett dylikt ytbeklädnadsmaterial". Sedan Rockwool International A/S (Rockwool) invänt mot patentet beslutade PRV genom det överklagade beslutet att patentet skulle upprätthållas i ändrad lydelse. PRV fann i sitt beslut att uppfinningen enligt patentkraven i ändrad lydelse uppfyllde patenterbarhetsvillkoren.

I målet har Rockwool International anfört dokumenten D1-D15:

D1 WO 03008854 A2

D2 GB 2108046 A

D3 US 3837992 A

D4 US 4271218 A

D5 WO 9637728 A1

D6 US 6635322 B1

D7 Ullmann's Encyclopedia of industrial chemistry, vol. A20, p. 494

D8 EP 0394117 B1

D9 utdrag från hemsida <http://en.wikipedia.org/wiki/polyester> (bilaga 2)

D10 datablad från Tscheulin-Rothal, daterat september 1988 (bilaga 3)

D11 datablad från Tscheulin-Rothal, daterat augusti 1990 (bilaga 4)

D12 utdrag från hemsida [http://en.wikipedia.org/wiki/polvethylene\\_terephthalate](http://en.wikipedia.org/wiki/polvethylene_terephthalate) (bilaga 5)

D13 utdrag från hemsida <http://www.chemvigi.com/pdf/ASTMD36-95.pdf> (bilaga 6)

D14 US 4447490 A

D15 WO 02/055801 A1

**Uppfinningen**

Av patentets beskrivning framgår bland annat följande om uppfinningen och dess bakgrund.

Uppfinningen avser ett ytbeklädnadsmaterial i laminatform avsett för isoleringssystem för installationsdelar såsom rör, behållare och ventilationskanaler.

Uppfinningen avser även ett isoleringssystem innefattande ett sådant ytbeklädnadsmaterial.

Inom byggnads- och processtekniken är det mycket vanligt att isolera till exempel rör och behållare med rörskålar, skivor eller mattor. En typisk isolering består av ett termiskt isolerande material, exempelvis mineralull, som på den sida som vänds mot omgivningen har ett laminerat skyddande ytskikt av plast, papper eller metall. Det skyddande ytskiktet uppvisar av tradition en mycket ringa tjocklek, vilket gör ytskiktet känsligt för mekanisk åverkan som kan uppstå under exempelvis montering eller underhåll. I de fall det utgörs av aluminium används tjocklekar i storleksordningen 18  $\mu\text{m}$ , vilket anses vara den nedre gränsen för en diffusionstät aluminiumfolie. Ett vanligt sätt att öka ytskiktets hållfasthet är att förse det med en armering, exempelvis kan glastrådar integreras i ytskiktet. En sådan armering är emellertid inte tillräcklig för att ge isoleringens ytskikt erforderliga mekaniska egenskaper.

Då isoleringen utgörs av en rörskål uppvisar denna av monteringsmässiga skäl en slits som gör att den enkelt kan öppna upp och monteras på ett rör. Slitsen förseglas därefter med exempelvis en tejprensa. För ökade mekaniska egenskaper och säkerställande av fullgod försegling är det vanligt förekommande att dessa isoleringar vid montering kompletteras med ett utvändigt skal, vilka kan utgöras av styva, men fortfarande böjbara, formpressade plastkåpor eller metallkåpor.

Ett ändamål med uppfinningen är att tillhandahålla ett ytbeklädnadsmaterial i laminatform som uppvisar sådana mekaniska egenskaper, i synnerhet rivstyrka, slagtålighet och penetreringsstyrka, att det klarar normal hantering under montering, underhåll och i övrigt normala påfrestningar under isoleringens livstid.

Ett annat ändamål är att tillhandahålla ett ytbeklädnadsmaterial som klarar gällande brandkrav, exempelvis standarden enligt SS 02 48 23/NORDTEST-FIRE 004, "Ytskikt klass 1" (standard NORDTEST).

Ytterligare ett ändamål är att ytbeklädnads materialet ska kunna appliceras på ett isoleringssystem under framställning av detta för att bilda en integrerad enhet.

Ytbeklädningsmaterialet ska vidare vara enkelt och billigt att tillverka.

Det ska dessutom med ringa tjocklek vara smidigt och lätt att hantera samtidigt som det uppvisar ovan nämnda egenskaper.

Ännu ett ändamål är att ett isoleringssystem med detta ytbeklädnadsmaterial ska kunna monteras direkt på plats utan ytterligare inklädnad i form av exempelvis separata kåpor.

Dessa ändamål uppnås med ett ytbeklädnadsmaterial vilket innefattar ett första utvändigt metallfolieskikt, exempelvis aluminiumfolie, med en tjocklek inom intervallet 20-100  $\mu\text{m}$ , samt ett plastskikt som är anordnat mellan det första utvändiga metallfolieskiktet och isoleringssystemets yta, vilket plastskikt har en tjocklek inom intervallet 10-100  $\mu\text{m}$ , ett termoplastiskt bindemedelsskikt avsett för förbindning av nämnda ytbeklädnadsmaterial mot nämnda isoleringssystem, varvid smältpunkten hos det termoplastiska bindemedelsskiktet understiger smältpunkten hos nämnda plastskikt samt ett andra invändigt metallfolieskikt, exempelvis aluminiumfolie, med en tjocklek inom intervallet 5-30  $\mu\text{m}$ , vilket metallfolieskikt är anordnat mellan nämnda plastskikt och nämnda termoplastiska bindemedelsskikt.

De mekaniska egenskaperna erhålls primärt av plastskiktet medan brandtåligheten erhålls av det utvändiga metallfolieskiktet. Genom att smältpunkten hos det termoplastiska bindemedelsskiktet, avsett för förbindning av ytbeklädnadsmaterialet mot ett isoleringssystem, understiger smältpunkten hos nämnda plastskikt påverkas inte plastskiktet och därmed ytbeklädnadsmaterialets invändiga struktur vid värmeapplicering av ytbeklädnadsmaterialet. Det invändiga metallfolieskiktet är avsett att samverka med plastskiktet och bidra med ökade mekaniska egenskaper och brandegenskaper.

### **Yrkanden**

Rockwool har yrkat att patentet ska upphävas i sin helhet.



Isover har yrkat att patentet ska upprätthållas i sin nuvarande lydelse med patentkrav inkomna den 14 oktober 2009.

Uppfinningen definieras i de självständiga kraven 1 och 11 på följande sätt:

1. Ytbeklädnadsmaterial (1) i laminatform avsett för isoleringssystem (7) för installationsdelar såsom rör, behållare, ventilationskanaler, vilket isoleringssystem (7) uppvisar en yta som ytbeklädnadsmaterialiet (1) är avsett att klä innefattande ett första utvändigt metallfolieskikt (3), exempelvis av aluminiumfolie, med en tjocklek inom intervallet från 20 - 100  $\mu\text{m}$  och mest föredraget 30 - 60  $\mu\text{m}$ , ett plastskikt (4) som är anordnat mellan det första metallfolieskiktet (3) och isoleringssystemets (7) yta, vilket plastskikt har en tjocklek inom intervallet 10 - 100  $\mu\text{m}$  och mest föredraget 20 - 40  $\mu\text{m}$ , ett termoplastiskt bindemedelsskikt (5) som är avsett för förbindning av nämnda ytbeklädnadsmaterial (1) mot nämnda isoleringssystem (7), varvid smältpunkten hos nämnda termoplastiska bindemedelsskikt (5) understiger smältpunkten hos nämnda plastskikt (4), samt ett andra invändigt metallfolieskikt (6), exempelvis aluminiumfolie, vilket uppvisar en tjocklek inom intervallet 5 - 30  $\mu\text{m}$  och mest föredraget 7 - 20  $\mu\text{m}$ , vilket metallfolieskikt är anordnat mellan nämnda plastskikt (4) och nämnda termoplastiska bindemedelsskikt (5).

11. Isoleringssystem för installationsdelar såsom rör, behållare och ventilationskanaler, vilket isoleringssystem (7) uppvisar ett termiskt isolerande material (9) med en yta som ett ytbeklädnadsmaterial (1) är avsett att klä, vilket ytbeklädnadsmaterial (1) är i laminatform och innefattar ett första utvändigt metallfolieskikt (3), exempelvis av aluminiumfolie, med en tjocklek inom intervallet 20 - 100  $\mu\text{m}$  och mest föredraget 30 - 60  $\mu\text{m}$ , ett plastskikt (4) som är anordnad mellan det första metallfolieskiktet (3) och det termiskt isolerande materialets (9) yta, vilket plastskikt (4) har en tjocklek inom intervallet 10 - 100  $\mu\text{m}$  och mest föredraget 20 - 40  $\mu\text{m}$ , ett termoplastiskt bindemedelsskikt (5) som är avsett för förbindning av nämnda ytbeklädnadsmaterial (1) mot nämnda termiskt isolerande material (9), varvid smältpunkten hos nämnda termoplastiska bindemedelsskikt (5) understiger smältpunkten hos nämnda plastskikt (4), samt ett andra invändigt metallfolieskikt (6), exempelvis aluminiumfolie, vilket uppvisar en tjocklek inom intervallet 5 - 30  $\mu\text{m}$  och mest föredraget 7 - 20  $\mu\text{m}$ , vilket metall-

folieskikt är anordnat mellan nämnda plastskikt (4) och nämnda termoplastiska bindemedelsskikt (5),  
varvid isoleringssystemet innefattar en av nämnda ytbeklädnadsmaterial (1) anordnad flik (11) för applicering över en skarv mellan två intilliggande isoleringssystem (7) eller en slits (10) i nämnda isoleringssystem.

## **Grunder**

Rockwool har som grund för sin talan anfört att uppfinningen saknar uppfinningshöjd.

Isover har som grund för sin talan anfört att uppfinningen har uppfinningshöjd.

## **Utveckling av talan**

Parterna har till utveckling av talan anfört bland annat följande.

### Rockwool

Krav 1 saknar uppfinningshöjd med dokument D8 utgörande den närmaste kända teknik. Detta påvisas under särskild hänvisning till dokumenten D1, D2, D4, D10 och D11.

I D8 visas ett ytbeklädnadsmaterial som har en laminatform och är avsett att bekläda ett isoleringssystem. Ytbeklädnads materialet visas i figur 1 och omfattar ett multiskikt ("feuille multicouche") i form av skikten aluminium/polyester/aluminium ("triplex aluminium/polyester/aluminium"). Laminatet förbinds med isoleringssystemet med hjälp av ett termoplastiskt bindemedel.

Vid den muntliga förhandlingen påpekade bolaget även följande angående nämnda multiskikt. Laminatet i D8 är sammansatt av två olika material, varav det ena är ett multiskikt. Fackmannen ser multiskiktet som en separat enhet. Det har vid något tillfälle utgjort ett eget skikt. Eftersom materialen slagits samman är det därför rimligt att fackmannen åter kan separera dem. Han erhåller då nämnda multiskikt.

I D8 nämns angående tidigare känd teknik att det är känt att använda ytbeklädnadsmaterial bestående av ett aluminiumskikt och ett plastskikt som isolering och att detta ger ett bra mekaniskt skydd. I D8 beskrivs att man vill öka den mekaniska styrkan hos det kända ytbeklädnads materialet genom att tillföra ett invändigt aluminiumskikt. Då bildas ett multiskikt med ett första utvändigt metallfolieskikt, ett mellanliggande plastskikt och ett ytterligare invändigt metallfolieskikt. På ytbeklädnads materialets utvändiga ovanyta är ett bitumensskikt påfört, vars syfte är att skydda ytbeklädnanden mot UV-ljus. Uppfinnaren i D8 är medveten om att brandregler ska observeras, eftersom det specificeras att ett svårantändligt bitumenmaterial ska användas ("solutions de bitumes non inflammable"). Sammanfattningsvis konstateras att D8 avser ett ytbeklädnadsmaterial med särskilt goda mekaniska och brandmässiga egenskaper. D8 uttalar sig inte om tjocklek för ett enskilt materialskikt men nämner att den totala tjockleken för de tre skikt som utgör multiskiktet kan vara 30-50 µm.

Patentkrav 1 är nytt i förhållande till D8 eftersom tjocklekarna av metallskikten och plastskiktet inte uttryckligen föreslås.

Det objektiva tekniska problemet ska baserat på D8 formuleras enligt följande: "Hur kan man uppnå bättre mekaniska egenskaper, samtidigt som man uppfyller gällande brandkrav?".

Enligt patentets krav 1 krävs ingen särskild relation mellan skiktens tjocklekar för att lösa det objektiva problemet som uppfinningen adresserar. Patentkrav 1 klargör att man med en godtycklig kombination av tjocklekar inom angivna intervall vill kunna uppnå goda mekaniska egenskaper och uppfylla gällande brandkrav. De i patentkravet angivna tjockleksintervallerna gör att det kan röra sig om allt från tunna till tjocka skikt. Till stöd för effekten har patenthavaren i patentets beskrivning redovisat ett test där endast en sammansättning av skikten har testats. I samband med uppfinningshöjdsbedömning kan ifrågasättas om den tekniska effekten gör sig gällande med alla kombinationer av tjocklekar inom de intervall som anges i patentkrav 1.

De tjockleksintervall som anges i patentkrav 1 ligger inom vad en fackman rutinmässigt arbetar med och därmed vad han omedelbart vill

kunna föreslå, om han med utgångspunkt från D8 ska lösa det objektiva problemet.

D2 och D4 visar tillsammans med D10 och D11, att fackmannen inom teknikområdet i allmänhet arbetar med tjocklekar av plastskikt och aluminiumfolier inom relativt stora intervaller.

I D2 beskrivs att det är allmänt känt att använda en metallfolie på en plastfolie och där föreslås användning av metallfolier med en tjocklek av 10 - 70  $\mu\text{m}$  och plastskikt med en tjocklek av 100  $\mu\text{m}$ . Av D4 framgår att man till ytbeklädnader för isoleringssystem kan använda metallfolier med en tjocklek inom intervallet 10-40  $\mu\text{m}$  och plastlager med en tjocklek i intervallet 20-50  $\mu\text{m}$ . Fackmannen har således arbetat med aluminiumfolier med tjocklekar i intervallet 10 -100  $\mu\text{m}$ , och plastskikt med tjocklekar i intervallet 20-100  $\mu\text{m}$  lång tid före prioritetsdatumet för patentet.

Vidare framgår av D10 och D11 att det sedan 1990 marknadsförts två olika laminat, av typ ”triplex aluminium/polyester/polyester”, som används för att bekläda isoleringssystem och som uppfyller brandreglerna enligt norm DIN4102. Dessa ytbeklädnadsmaterial förbinds med isoleringsmaterial och omfattar två skikt av en aluminiumfolie med ett mellanliggande plastskikt. I D10 ingår ett mellanliggande plastskikt i form av PU-lim. D10 och D11 anger tjocklekar hos aluminiumfolien i storleksordningen 20 respektive 12  $\mu\text{m}$ .

Mot bakgrund av ovanstående anger krav 1 enbart det som en fackman rutinmässigt föreslår om han ska lösa det uppställda problemet.

Vidare visar D8, D1 och D4 i kombination att det är närliggande för fackmannen att komma fram till såväl önskade mekaniska egenskaper som de egenskaper som klarar gällande brandkrav. Från D1 är en ytbeklädnad med metallfolieskikt och plastskikt redan känd med en sådan uppbyggnad att den kan godkännas enligt standard NORDTEST. Särskilt hänvisas till tabellen på sidan 8, där varje rad utgör ett exempel, och till första rutan i tabellen där det framgår att skiktet är metalliserad polyeten. Problemet med att uppfylla gällande brandkrav är därför redan löst genom D1. D4 visar att fackmannen vid isoleringsmaterial av detta slag arbetar med aluminiumfolier med en tjocklek av 10-40  $\mu\text{m}$  och plast-

skikt med en tjocklek av 20-50  $\mu\text{m}$ , vilket uppges resultera i ett laminat med goda mekaniska egenskaper. Mot bakgrund av detta anger krav 1 enbart det som fackmannen som utgår från D8 och har kännedom om D1 och D4 rutinmässigt föreslår om han ska lösa det uppställda problemet.

Av dessa skäl uppvisar krav 1 inte någon uppfinningshöjd.

Beträffande patentkrav 11 avseende ett isoleringssystem kan det objektiva problemet formuleras som: "Hur kan man uppnå ett isoleringssystem med en ytbeklädnad, som är enkel att montera på t.ex. ett rör och som har bättre mekaniska egenskaper, samtidigt som man uppfyller gällande brandkrav?"

Lösningen på detta problem anknyter till utformningen av själva ytbeklädnaden enligt krav 1; användningen av en flik gör det enkelt att montera isoleringssystemet på ett rör, och har i realiteten inget att göra med ytbeklädnadens mekaniska- eller brandmässiga egenskaper.

Enligt tidigare resonemang anger krav 1 inte något som innefattar uppfinningshöjd med utgångspunkt i D8 som närmast kända teknik med beaktande av tekniken i D1, D2, D4, D10 och D11. Ytbeklädnaden i D8 har en flik varvid isoleringssystemet är enkelt att montera på ett rör. Som utgångspunkt löser D8 därmed båda de ovan uppställda problemen. Också D4 har en flik och användningen av en flik för att underlätta montering av isoleringssystemet är känt för fackmannen, jmf D1, D5 och D6.

Krav 11 saknar således också uppfinningshöjd.

### Isover

Dokument D8 får anses utgöra närmast känd teknik. Det visar ett ytbeklädnadsmaterial med ett antal skikt innefattande ett första yttre bitumenskikt, ett andra fiberbaserat förstärkningsskikt samt ett tredje, inre multilagerskikt. Bitumenskiktet, förstärkningsskiktet och multilagerskiktet samverkar för att åstadkomma ett ytbeklädnadsmaterial som ger ett effektivt skydd mot nötning, ånga/vatten, perforation och stötar. I D8 nämns inget om att materialet ska klara någon brandskyddsstandard men där uppges att bitumenskiktet bidrar med flamskydd. De stora skill-

naderna mellan laminatet enligt D8 och ytbeklädnads materialet enligt krav 1 är olika antal skikt, typen av skikt och att laminatet enligt uppfinningen har metall ytterst.

Den tekniska effekten av skillnaderna ovan är erhållande av ett alternativt material med bra eller snarare förbättrade brandegenskaper.

Det objektiva problemet kan formuleras såsom att åstadkomma ett alternativt ytbeklädnads material som klarar uppsatta brandkrav men även uppvisar god mekanisk styrka.

Uppfinningen enligt patentet ger lösningen på problemet genom kombinationen av ett utvändigt metallfolieskikt, ett plastskikt och ett invändigt metallfolieskikt där respektive skikt har de angivna tjocklekarna.

Vad gäller ytbeklädnads materialens uppbyggnad finns grundläggande materiella skillnader mellan det ytbeklädnads material som visas i D8 och uppfinningen. Skillnaderna ligger bland annat i antalet skikt där D8 innehåller sex skikt medan uppfinningen enligt krav 1 bara innehåller tre stycken. Det multilagerskikt som ingår i D8 har heller inte som uppgift att skydda mot brand.

Beträffande godstjocklekar har fackmannen ingen anledning att söka stöd för valet av godstjocklekar i det multilagerskikt som ingår som en invändig komponent i D8. Resonemanget bygger på ett antagande att fackmannen med D8 som utgångspunkt skulle skala bort de två yttersta skikten och enbart bibehålla det tredje inre multilagerskiktet som därmed skulle bilda det yttersta skiktet i laminatet. Fackmannen finner inga som helst pekare i D8 till att reducera antalet skikt, och därvid plocka bort de två skikt som uttalat bidrar till lösningen av det objektiva problemet, nämligen bitumensskiktet (brand) och det fiberbaserade skiktet (stötär och deformation, dvs. en form av mekanisk styrka).

Uppfinningen enligt krav 1 uppvisar uppfinningshöjd gentemot D8 enskilt.

*D8 i kombination med D1*

D1 nämner inget om brand, vilket påstås av Rockwool i sitt överklagande. Materialet har en annan struktur än den som omnämns i såväl D8 som i uppfinningen enligt krav 1, nämligen åtminstone ett skikt av termoplastisk polymer och ett valfritt aluminiumskikt vars huvudsyfte är att ge en metalliserad yta. Aluminiumskiktet kan vara anordnat antingen utvändigt eller invändigt och kan antingen vara en film eller en metalliserad yta.

De strukturella skillnaderna mellan D8 eller D1 och uppfinningen enligt krav 1 är så stora att den enda rimliga slutsatsen är att kombinationen av tekniken i D8 och D1 är orimlig och att uppfinningen enligt kravet 1 därför uppvisar uppfinningshöjd.

*D8 i kombination med D2*

D2 beskriver ett ytbeklädnadsmaterial som klarar samma brandklassning som uppfinningen enligt krav 1. Materialet har dock en annan struktur. Det har ett utvändigt metallfolieskikt (aluminium) med syfte att agera diffusionsspärr och skydda mot strålningsförluster och ett invändigt substrat av polyolefin med mineralbaserat fyllmedel, där substratet ger mekanisk styrka och brandmotstånd. För att öka flamskyddet kan det finnas ett utvändigt lackskikt.

Om fackmannen utgår från D8 och vänder sig till D2 skulle han inse att där finns ett material som uppfyller rätt brandklass men detta åstadkoms med en helt annan struktur än i föreliggande uppfinning, d.v.s. med en invändig stomme av polyolefin med mineralbaserat fyllmedel. Om fackmannen skulle vilja förbättra brandskyddet skulle han laborera med mängden fyllmedel alternativt med det lackskikt som beskrivs. Detta skulle resultera i ett laminat som har förutsättningar att uppfylla det uppställda brandkravet men ha en helt annan struktur än den som anges i kravet 1.

Uppfinningen enligt krav 1 uppvisar såldes uppfinningshöjd gentemot kombinationen D8 och D2.

*D8 i kombination med D4*

D4 nämner inte något om brandegenskaper och har en annan struktur än den som beskrivs i D8, nämligen består strukturen i D4 av plast, metall, plast, alternativt metall, plast. Materialet saknar vidare ett bindemedels-skikt.

Om fackmannen vill öka den mekaniska styrkan skulle han med ledning av D4 anordna ett utvändigt plastskikt. Han får däremot ingen hjälp avseende frågan om brand. Slutsatsen är därför att uppfinningen enligt kravet 1 har uppfinningshöjd relativt kombinationen av tekniken i D8 och D4.

*D8 i kombination med D10*

D10 är ett datablad som beskriver ett laminat innehållande fyra skikt.

Det framgår klart av D10 att den mekaniska styrkan i materialet skapas av ett skikt med glasfibertrådar som är anordnat mellan metallskikten. Laminerade glastrådar ger dock inte laminatet erforderliga mekaniska egenskaper och i synnerhet ingen slagåtlighet. Vidare limmas glastrådarna i D10 fast i aluminiumskikten med PU-lim (4g/m<sup>2</sup>). Ett skikt med denna mycket begränsade tjocklek kan inte likställas med uppfinningens plastskikt. D10 saknar således ett plastskikt som är anordnat mellan de två metallfolieskikten. Såsom klart framgår i föreliggande patent erhålls med ett sådant plastskikt en seghet i ytbeklädnadsmaterialet, som ger både rivstyrka och penetreringsstyrka. Att fackmannen utifrån dessa strukturella skillnader dessutom skulle dra några slutsatser om materialtjocklekar hos de ingående skikten får anses osannolikt.

Av D10 framgår att materialet där i kombination med mineral eller glasull, till exempel stenullsmattor, är icke brännbart enligt standard DIN 4102 A 2. Detta motsvarar inte den för uppfinningen relevanta standarden NORDTEST. Man kan inte jämföra olika standarder och i synnerhet inte när man har olika bedömningskriterier. Att fackmannen skulle studera D10 för att finna vägledning vid valet av tjocklekar för att åstadkomma ett laminat som uppvisar goda mekaniska egenskaper och som samtidigt uppfyller kraven enligt "Ytskikt klass 1" enligt nämnda standard NORDTEST är långsökt då D10 inte alls diskuterar mekanisk styrka och dessutom har en annan strukturell uppbyggnad.



Den strukturella uppbyggnaden på laminatet i D10 talar därför mot att fackmannen skulle beakta detta dokument vid valet av tjocklekar och/eller uppbyggnad av ett material i enlighet med föreliggande uppfinning.

Slutsatsen är därför att uppfinningen enligt krav 1 uppvisar uppfinningshöjd gentemot kombinationen av D8 och D10.

#### *D8 i kombination med D11*

D11 är ett datablad som beskriver ett laminat innehållande fem skikt, med början utifrån ett svårantändligt färgskikt, ett flexibelt aluminiumskikt (tjocklek 0,020 mm), ett specialskikt av okänt slag, ett armerings-skikt av glastrådar och ett flexibelt aluminiumskikt (tjocklek 0,012 mm). D11 saknar relevans eftersom det beskrivna laminatet uppvisar en struktur som saknar likheter med den som anges i uppfinningens krav 1, dvs. med början utifrån består av aluminium, plast, aluminium och bindemedel.

Av D11 framgår att laminatet limmat mot dämpande plattor av mineralfiber klarar brandkraven enligt standard DIN 4102 B 1. Indexet B1 förefaller avse material som är svårantändliga och material som ofta är självsläckande, vilket ska jämföras med "Ytskikt klass 1" där man tittar på medeltemperatur och rökbildning. Olika standarder kan inte jämföras när bedömningskriterierna är olika.

Att fackmannen skulle studera D11 för att finna vägledning vid valet av tjocklekar för att åstadkomma ett material som uppvisar goda mekaniska egenskaper och samtidigt uppfyller kraven enligt standard NORDTEST är långsökt och inte rimligt med tanke på att D11 inte alls diskuterar mekanisk styrka.

Slutsatsen är därför att uppfinningen enligt krav 1 uppvisar uppfinningshöjd gentemot kombinationen av D8 och D11.

#### *D8 i kombination med D1 och D4*

Kombinationen av D8 med D1 och D4 bygger på en ex post facto analys, dvs. att man redan känner uppfinningen. Fackmannen har ingen anled-

ning att utföra denna kombination och kravet 1 har därför uppfinningshöjd i förhållande till denna teknik.

Eftersom uppfinningen enligt krav 1 anses uppfylla villkoren för patentbarhet gäller detta även uppfinningen enligt kravet 11.

I målet har hållits muntlig förhandling.

## DOMSKÄL

Patentbesvärslätten konstaterar att uppfinningen är ny, och gör följande bedömning av patentkraven med avseende på uppfinningshöjd.

Föreliggande uppfinning avser ett ytbeklädnadsmaterial i laminatform som besitter sådana mekaniska egenskaper att det klarar normala påfrestningar under isoleringens livstid. Ett annat ändamål är att tillhandahålla ett ytbeklädnadsmaterial som klarar gällande brandkrav.

Lösningen utgörs av ett ytbeklädnadsmaterial i enlighet med patentkrav 1.

Patentbesvärslätten anser, liksom Rockwool och Isover, att av de i målen anförda dokumenten får D8 anses representera den teknik som kommer uppfinningen enligt patentet närmast. Dokument D8 beskriver ett ytbeläggingsmaterial med flexibla egenskaper, som gör det lämpligt att isolera element av olika form, såsom till exempel rör. Ytbeläggingsmaterialet kan vara uppbyggt av ett yttre skikt av bitumen, följt av ett förstärkningsskikt bildat av ett ark av glasfiber, syntetfiber eller naturliga fibrer, ett därpå anordnat multilagerskikt samt adhesiv tejp längs kanten. Multilagerskiktet kan innefatta polyesterfilm limmad mellan två aluminiumfilmer. I D8 anges inget om tjockleken hos de enskilda filmskikten men den totala tjockleken hos multilagerskiktet anges till 30-50 µm.

I D8 anges de olika skikten bidra med följande egenskaper. Bitumenskiktet ger UV- och åldringsskydd, nötningstålighet och förhindrar antändning. Förstärkningsskiktet ska skydda mot deformation och slag (stötar)

och multilagerskiktet ska ge laminatet god förmåga att motstå inträngning av vatten och ånga, penetrering samt en god rivstyrka.

Rockwool har framfört att patentets krav 1 inte specificerar antalet skikt. Patentbesvärslagen delar denna uppfattning, vilken också stöds av patentets beskrivning sid. 9 som anger att materialet kan innefatta ytterligare skikt. Dock är det Patentbesvärslagens mening att kravet anger att ytbeklädnadsmaterialets yttersta skikt utgörs av ett metallfolieskikt.

Uppfinningen enligt patentkrav 1 skiljer sig från tekniken enligt D8 främst genom att det utvändiga skiktet utgörs av ett metallfolieskikt och genom att tjockleken hos de ingående skikten av metallfolie och hos plastskiktet definieras.

Såsom framfördes vid den muntliga förhandlingen ifrågasätter Rockwool om ett laminat enligt patentkrav 1 med alla de möjliga kombinationer av tjocklekar hos de olika skikten har angiven teknisk effekt, det vill säga om alla dessa kombinationer uppfyller gällande brandkrav och har goda mekaniska egenskaper.

Enligt rättens mening finns det dock ingen anledning att ifrågasätta denna tekniska effekt hos det patenterade ytbeklädnadsmaterialet.

Då laminatet i D8 är utformat för att klara mekaniska påfrestningar och det yttre skiktet, bitumenskiktet, åtminstone i viss utsträckning bidrar med brandskydd får fackmannen med utgångspunkt i D8 anses vara ställd inför problemet att tillhandahålla ett alternativt ytbeklädnads-material som motstår brand och har goda mekaniska egenskaper.

Rockwool har framfört att multilagerskiktet i D8 ska betraktas som en separat enhet. Bolaget hävdar att det i D8 beskrivna laminatet har satts samman av två olika delar, varav ett utgörs av detta multilagerskikt. Därmed skulle det enligt Rockwool ligga nära till hands för fackmannen att separera dem. Multilagerskiktet har som yttersta skikt en aluminiumfolie. Beträffande vad som ska utgöra laminatets yttersta skikt har Rockwool i detta sammanhang även framfört att fackmannen av D1 (se

spec. tabellen sid. 8) men även från annan känd teknik får informationen om att ett metallskikt är lämpligt att placera ytterst.

Enligt Patentbesvärslättens mening skulle fackmannen trots att kända laminat för ytbeklädnader kan ha ett metallfolieskikt ytterst inte utifrån ovanstående känd teknik finna något som föranleder honom att separera laminatet i D8 och på så vis erhålla nämnda multilagerskikt.

Eftersom laminatet i D8 redan bidrar till lösningen av det aktuella problemet, det vill säga att tillhandahålla ett ytbeklädnadsmaterial som motstår brand (genom bitumenskiktet) och har goda mekaniska egenskaper (bl. a. genom förstärkningsskiktet) saknar fackmannen ställd inför det aktuella problemet, enligt rättens mening, incitament att separera det i D8 kända laminatet.

Enbart en sådan separering leder heller inte fram till uppfinningen enligt patentkrav 1 utan fackmannen skulle därefter behöva använda en av laminatets separerade delar, multilagerskiktet, som en utgångspunkt för vidare dimensioneringsarbete.

För att exemplifiera vilka tjockleksintervaller en fackman inom ytbeklädnadsområdet rutinmässigt arbetar med har Rockwool hänvisat till den genom D2, D10 och D11 kända tekniken.

Produktbladen D10 och D11 samt dokument D2 visar dels hur laminat med ingående skikt av aluminiumfolie kan vara uppbyggda, dels mått på tjockleken för vissa enskilda skikt som i viss mån är i överensstämmelse med de tjocklekar som enskilda skikt i uppfinningen enligt patentkrav 1 anges ha. För att uppnå brandsäkerhet ingår dock aluminiumfolien i kombinationer med andra material än i laminatet enligt uppfinningen, något som snarare får anses leda fackmannen bort från den i patentkravet 1 angivna lösningen. Det i D11 visade laminatet innehåller ett första yttre svårantändligt färgskikt, ett flexibelt aluminiumskikt, ett specialskikt, ett armeringsskikt och ett aluminiumskikt. Deras placering och hur de kombinerats med andra materials skiljer sig dock i flera avseenden från uppfinningen. I D2 beskrivs ett laminat för isolering av rör där ett substrat av polymerbaserat material försetts med ett diffusionstätt lager (lämpligen aluminium) och där brandhårdighet uppnås

antingen genom att ett fyllmedel tillsätts substratet eller genom att metallfolien förses med ett lacklager. Denna kända teknik anvisar således inte fackmannen alternativet att de önskade egenskaperna kan erhållas med en sådan kombination av material och tjocklekar som uppfinningen inbegriper.

Följaktligen ger inte heller den i de anförda dokumenten D10, D11 eller D2 kända tekniken fackmannen någon ledning till att modifiera ytbeklädnads materialet i D8 till överensstämmelse med det i patentkravet 1 definierade ytbeklädnads materialet.

Rockwool har vidare framfört att fackmannen som har D8 som utgångspunkt skulle konsultera D4 för att lösa problemet med mekanisk hållfasthet och D1 för att hitta en lösning på problemet att klara gällande brandkrav. Genom D4 känner fackmannen till att laminat med goda mekaniska egenskaper kan sättas samman av metallfolieskikt och plastskikt samt får information om i sammanhanget användbara tjockleksintervaller, tjocklek hos metallfolie av 10-40  $\mu\text{m}$  och hos plastskikt av 20-50  $\mu\text{m}$ . D1 anger inget om brandskyddsaspekter men exemplifierar att en metallfolie kan placeras ytterst i ett ytbeklädnadslaminat. Det är rättens uppfattning att fackmannen som utgår från det ovan redovisade laminatet i D8 och ska lösa det aktuella problemet inte finner några anvisningar vare sig i D8, D4 eller D1 till att de avsedda effekterna kan uppnås genom att utgående från ytbeläggningss materialet i D8 utvälja multilager-skiktet och därefter modifiera detta skikt till en sådan utformning som anges i patentkravet 1.

Även om fackmannen med tekniken i D8 som utgångspunkt, ställd inför det aktuella problemet, skulle studera de olika materialen, deras sammansättningar i olika laminat och föreslagna tjocklekar i den teknik som anförts skulle han inte få någon ledning till att ta fram ett ytbeklädnads material med den i patentkravet 1 definierade kombinationen av material och tjocklekar.

Patentbesvärsträtten kan inte heller finna att något som framgår av den anförda tekniken eller vad som i sammanhanget får anses vara allmänt känt skulle ge fackmannen sådan ledning att han ställd inför det aktuella

problemet skulle modifiera känd teknik på sådant sätt att den överensstämmer med uppfinningen enligt patentkrav 1.

Uppfinningen enligt patentkrav 1 anses därför ha uppfinningshöjd.

I enlighet med det resonemang som förts ovan för patentkravet 1 bedöms inte heller isoleringssystemet enligt patentkrav 11, i vilket ytbeklädnads materialet enligt krav 1 ingår, vara närliggande för fackmannen.

Vid denna bedömning ska överklagandet avslås och patentet upprätthållas.

**ANVISNING FÖR ÖVERKLAGANDE**, se bilaga 7 (Formulär A)

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I avgörandet har deltagit patenträttsråden Jeanette Bäckvall, ordförande, Yvonne Siösteen och Heléne Eliasson, referent. Enhälligt

PATENTBESVÄRSRÄTTEN	
Ink	2010 -08- 1.3
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D9

# Polyester

From Wikipedia, the free encyclopedia

**Polyester** is a category of polymers which contain the ester functional group in their main chain. Although there are many polyesters, the term "polyester" as a specific material most commonly refers to polyethyleneterephthalate (PET). Polyesters include naturally-occurring chemicals, such as in the cutin of plant cuticles, as well as synthetics through step-growth polymerization such as polycarbonate and polybutyrate. Natural polyesters and a few synthetic ones are biodegradable, but most synthetic polyesters are not.

Depending on the chemical structure polyester can be a thermoplastic or thermoset, however the most common polyesters are thermoplastics.<sup>[1]</sup>

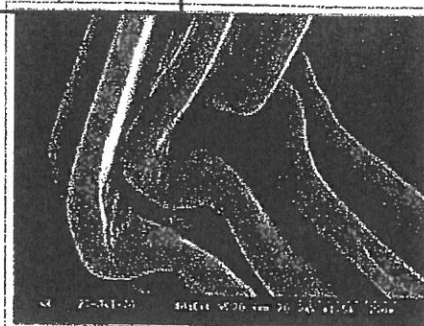
Fabrics woven from polyester thread or yarn are used extensively in apparel and home furnishings, from shirts and pants to jackets and hats, bed sheets, blankets and upholstered furniture. Industrial polyester fibers, yarns and ropes are used in tyre reinforcements, fabrics for conveyor belts, safety belts, coated fabrics and plastic reinforcements with high-energy absorption. Polyester fiber is used as cushioning and insulating material in pillows, comforters and upholstery padding.

While synthetic clothing in general is perceived by some as having a less-natural feel compared to fabrics woven from natural fibres (such as cotton and wool), polyester fabrics can provide specific advantages over natural fabrics, such as improved wrinkle resistance. As a result, polyester fibres are sometimes spun together with natural fibres to produce a cloth with blended properties. Synthetic fibres also can create materials with superior water, wind and environmental resistance compared to plant-derived fibres.

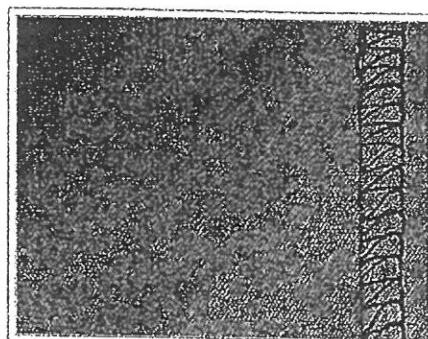
Polyesters are also used to make "plastic" bottles, films, tarpaulin, canoes, liquid crystal displays, holograms, filters, dielectric film for capacitors, film insulation for wire and insulating tapes.

Liquid crystalline polyesters are among the first industrially-used liquid crystalline polymers. They are used for their mechanical properties and heat-resistance. These traits are also important in their application as an abrasable seal in jet engines.

Polyesters are widely used as a finish on high-quality wood products such as guitars, pianos and vehicle / yacht interiors. Burns Guitars, Rolls Royce and Sunseeker are a few companies that use polyesters to finish their products. Thixotropic properties of spray-applicable polyesters make them ideal for use on open-grain timbers, as they can quickly fill wood grain, with a high-build film thickness per coat. Cured polyesters can be sanded and polished to a high-gloss, durable finish.



SEM picture of a bend in a high-surface area polyester fiber with a seven-lobed cross section



Close-up of a polyester shirt

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## Types

Polyesters as thermoplastics may change shape after the application of heat. While combustible at high temperatures, polyesters tend to shrink away from flames and self-extinguish upon ignition. Polyester fibres have high tenacity and E-modulus as well as low water absorption and minimal shrinkage in comparison with other industrial fibres.

Thermosetting polyesters are used as casting materials, and chemo setting polyester resins are used as fiberglass laminating resins and non-metallic auto-body fillers. Fibreglass-reinforced unsaturated polyesters find wide application in bodies of yachts and as body parts of cars.

According to the composition of their main chain, polyesters can be:

Composition of the main chain	Number of repeating units	Examples of polyesters	Examples of manufacturing methods
Aliphatic	Homopolymer	Polyglycolide or Polyglycolic acid (PGA)	Polycondensation of glycolic acid
		Poly(lactic acid) (PLA)	
	Copolymer	Polycaprolactone (PCL)	Ring-opening polymerization of caprolactone
		Polyethylene adipate (PEA)	
Semi-aromatic	Copolymer	Polyhydroxyalkanoate (PHA)	
		Polyethylene terephthalate (PET)	Polycondensation of terephthalic acid with ethylene glycol
		Polybutylene terephthalate (PBT)	Polycondensation of terephthalic acid with 2,3-butanediol
		Polytrimethylene terephthalate (PTT)	Polycondensation of terephthalic acid with 1,3-propanediol
		Polyethylene naphthalate	Polycondensation of ethylene glycol with one or more



		(PEN)	naphthalene dicarboxylic acids
Aromatic	Copolymer	Vectran	

Increasing the aromatic parts of polyesters increases their glass transition temperature, melting temperature, thermal stability, chemical stability...

Polyesters can also be telechelic oligomers like the polycaprolactone diol (PCL) and the polyethylene adipate diol (PEA). They are then used as prepolymers.

## Industry

### Basics

Polyester is a synthetic polymer made of purified terephthalic acid (PTA) or its dimethyl ester dimethyl terephthalate (DMT) and monoethylene glycol (MEG). With 18% market share of all plastic materials produced, it ranges third after polyethylene (33.5%) and polypropylene (19.5%).

The main raw materials are described as follows:

- **Purified Terephthalic Acid – PTA – CAS-No.: 100-21-0**

Synonym: 1,4 Benzenedicarboxylic acid,  
Sum formula; C<sub>6</sub>H<sub>4</sub>(COOH)<sub>2</sub> , mol weight: 166,13

- **Dimethylterephthalate – DMT- CAS-No: 120-61-6**

Synonym: 1,4 Benzenedicarboxylic acid dimethyl ester  
Sum formula C<sub>6</sub>H<sub>4</sub>(COOCH<sub>3</sub>)<sub>2</sub> , mol weight: 194,19

- **Mono Ethylene Glycol – MEG – CAS No.: 107-21-1**

Synonym: 1,2 Ethanediol  
Sum formula: C<sub>2</sub>H<sub>6</sub>O<sub>2</sub> , mol weight: 62,07

More information about polyester raw materials can be found for PTA <sup>[2]</sup>, DMT <sup>[3]</sup> and MEG <sup>[4]</sup>, at the webpage INCHEM "Chemical Safety Information from Intergovernmental Organizations".

To make a polymer of high molecular weight a catalyst is needed. The most common catalyst is antimony trioxide (or antimony tri acetate):

**Antimony trioxide – ATO – CAS-No.: 1309-64-4** Synonym: non, mol weight: 291,51 Sum formula: Sb<sub>2</sub>O<sub>3</sub>

In 2008 about 10 000 t Sb<sub>2</sub>O<sub>3</sub> were used to produce around 49 Mio t polyethylene terephthalate.

Polyester is described as follows:

**Polyethylene Terephthalate CAS-No.: 25038-59-9** Synonym / abbreviations: polyester, PET, PES  
Sum Formula: H-[C<sub>10</sub>H<sub>8</sub>O<sub>4</sub>]-<sub>n=60-120</sub> OH, mol unit weight: 192,17

There are several reasons for the importance of Polyester:

- The relatively easy accessible raw materials PTA or DMT and MEG
- The very well understood and described simple chemical process of polyester synthesis
- The low toxicity level of all raw materials and side products during polyester production and processing
- The possibility to produce PET in a closed loop at low emissions to the environment
- The outstanding mechanical and chemical properties of polyester
- The recyclability
- The wide variety of intermediate and final products made of polyester.

In table 1 the estimated world polyester production is shown. Main applications are textile polyester, bottle polyester resin, film polyester mainly for packaging and specialty polyesters for engineering plastics. According to this table, the world's total polyester production might exceed 50 million tons per annum before the year 2010.

Table 1: World polyester production

Product Type	Market size per year	
	2002 [Mio t/a]	2008 [Mio t/a]
Textile-PET	20	39
Resin, Bottle/A-PET	9	16
Film-PET	1.2	1.5
Special Polyester	1	2.5
<b>TOTAL</b>	31.2	49

### Raw material producer

The raw materials PTA, DMT, and MEG are mainly produced by large chemical companies which are sometimes integrated down to the crude oil refinery where *p*-Xylene is the base material to produce PTA and liquefied petroleum gas (LPG) is the base material to produce MEG.

Large PTA producers are for instance BP, Reliance, Sinopec, SK-Chemicals, Mitsui, and Eastman Chemicals. MEG production is in the hand of about 10 global players which are headed by MEGlobal a JV of DOW and PIC Kuwait followed by Sabic.

Among the world's largest polyester producers are the following companies:

Artenius, Advansa, DAK, DuPont, Eastman, Hyosung, Huvis, Indorama, Invista, Jiangsu Hengli Chemical Fiber, Jiangsu Sanfangxian Industry, M&G Group, Mitsui, Mitsubishi, Nan Ya Plastics, Reichhold, Reliance, Rongsheng, Sabic, Teijin, Toray, Trevira, Tuntex, Wellman, Yizheng Sinopec, and Zhejiang Hengi Polymerization.

### Polyester processing

After the first stage of polymer production in the melt phase, the product stream divides into two different application areas which are mainly textile applications and packaging applications. In figure 2 the main applications of textile and packaging polyester are listed.

Table 2: Textile and packaging polyester application list

POLYESTER-BASED POLYMER (MELT or PELLET)	

Textile	Packaging
Staple fiber (PSF)	Bottles for CSD, Water, Beer, Juice, Detergents
Filaments POY, DTY, FDY	A-PET Film
Technical yarn and tire cord	Thermoforming
Non-woven and spunbond	BO-PET Biaxial oriented Film
Mono-filament	Strapping

Abbreviations: PSF = Polyester Staple Fiber; POY = Partially Oriented Yarn; DTY = Draw Textured Yarn; FDY = Fully Drawn Yarn; CSD = Carbonated Soft Drink; A-PET = Amorphous Polyester Film; BO-PET = Biaxial Oriented Polyester Film;

A comparable small market segment (<< 1 Million t/a) of polyester is used to produce engineering plastics and masterbatch.

In order to produce the polyester melt with a high efficiency, high-output processing steps like staple fiber (50–300 t/d per spinning line) or POY /FDY (up to 600 t/d split into about 10 spinning machines) are meanwhile more and more horizontal, integrated, direct processes. This means the polymer melt is directly converted into the textile fibers or filaments without the common step of pelletizing. We are talking about full horizontal integration when polyester is produced at one site starting from crude oil or distillation products in the chain oil -> benzene -> PX -> PTA -> PET melt -> fiber / filament or bottle-grade resin. Such integrated processes are meanwhile established in more or less interrupted processes at one production site. Eastman Chemicals introduced at first the idea to close the chain from PX to PET resin with their so-called INTEGREGX process. The capacity of such horizontal, integrated productions sites is >1000 t/d and can easily reach 2500 t/d.

Besides the above mentioned large processing units to produce staple fiber or yarns, there are ten thousands of small and very small processing plants, so that one can estimate that polyester is processed and recycled in more than 10 000 plants around the globe. This is without counting all the companies involved in the supply industry, beginning with engineering and processing machines and ending with special additives, stabilizers and colors. This is a gigantic industry complex and it is still growing by 4–8% per annum, depending on the world region. Useful information about the polyester industry can be found under <sup>[5]</sup> where a “Who is Producing What in the Polyester Industry” is gradually being developed.

## Synthesis

Synthesis of polyesters is generally achieved by a polycondensation reaction. See "condensation reactions in polymer chemistry". The General equation for the reaction of a diol with a diacid is :

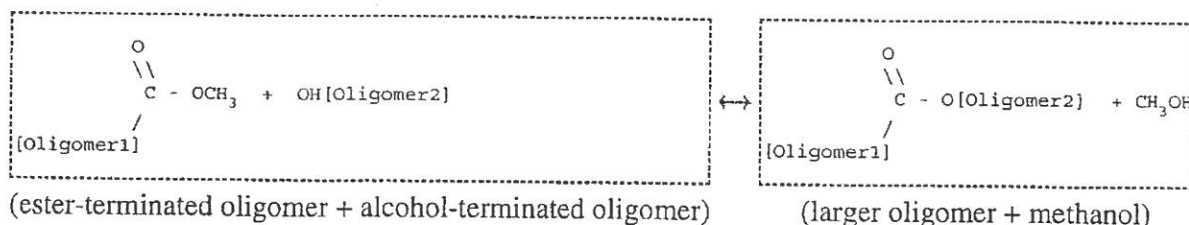
$$(n+1) R(OH)_2 + n R'(COOH)_2 \rightarrow HO[ROOCR'COO]_n ROH + 2n H_2O$$

### Azeotrope esterification

In this classical method, an alcohol and a carboxylic acid react to form a carboxylic ester. To assemble a polymer, the water formed by the reaction must be continually removed by azeotrope distillation.

### Alcoholic transesterification

*Main article: Transesterification*



### Acylation (HCl method)

The acid begins as an acid chloride, and thus the polycondensation proceeds with emission of hydrochloric acid (HCl) instead of water. This method can be carried out in solution or as an enamel.

#### Silyl method

In this variant of the HCl method, the carboxylic acid chloride is converted with the trimethyl silyl ether of the alcohol component and production of trimethyl silyl chloride is obtained

### Acetate method (esterification)

#### Silyl acetate method

### Ring-opening polymerization

Aliphatic polyesters can be assembled from lactones under very mild conditions, catalyzed anionically, cationically or metallorganically.

Thermosetting resins are generally copolymers of unsaturated polyesters with styrene. Polyester saturation is governed through the use of maleic acid or fumaric acid. In vinyl esters, saturation (or lack thereof) is found in the alcohol group of the polyester. The double bond of unsaturated polyester reacts with styrene resulting in a 3-D cross-linked structure. This structure acts as a thermoset. The cross-linking is initiated through an exothermic reaction involving an organic peroxide, such as methyl ethyl ketone peroxide or benzoyl peroxide.

## Health effects

A 1993 study found that polyester underwear reduced sperm count and sperm motility in male dogs. [6]

## Notes

- <sup>^</sup> Rosato, Dominick V.; Rosato, Donald V.; Rosato, Matthew V. (2004), *Plastic product material and process selection handbook*, Elsevier, p. 85, ISBN 9781856174312, <http://books.google.com/?id=Lqk5QgGoWFkC>.
- <sup>^</sup> PTA
- <sup>^</sup> DMT
- <sup>^</sup> MEG
- <sup>^</sup> Chemical Engineering – Polyester Information Platform
- <sup>^</sup> <http://dx.doi.org/10.1007/BF00296839>

## References

- *Textiles*, by Sara Kadolph and Anna Langford. 8th Edition, 1998.

## External links

- Polyester Market Polyester Market
- Chemical Engineering - Polyester Information Platform

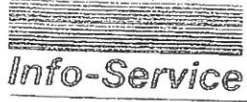
Retrieved from "http://en.wikipedia.org/wiki/Polyester"

Categories: Carboxylate esters | Packaging materials | Plastics | Polyesters | Synthetic resins | Synthetic fibers | Thermoplastics | Dielectrics | Airship technology | Recyclable materials | Thermosetting plastics

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Bilaga 3



Bauisolierungen  
Segment 81/08

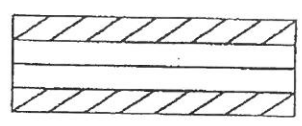
Flexible Packstoffe - Technische Verbunde

D10  
**Tscheulin-**  
**Rothal GmbH**  
Ein Unternehmen der  
VAV Europack Gruppe

<b>PATENTBESVÄRSRÄTTEN</b>	
Ink	2010 -08- 1.3
Mål nr	Aktbil
10-124	8

### Aluminium-Gelege-Aluminiumverbund

#### Materialaufbau:



- Aluminiumfolie 0,020 mm hart oder weich
- Glasfadengelege Verstärkung 12 x 12 mm
- Lackkaschierung PU-Kleber 4 g/qm
- Aluminiumfolie 0,012 mm weich

#### Lieferform: Rollen

#### Eigenschaften:

- In Verbindung mit Mineral-Glaswoll- bzw. Steinwoll-Matten unbrennbar nach DIN 4102 A 2.
- Wasserdampfdicht nach DIN 53 122.

#### Verwendungszweck:

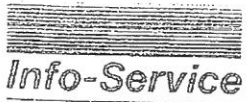
- Bauisolation im speziellen Deckschicht für Mineral-Glaswoll- bzw. Stein-Matten bzw. -Platten.

#### Bemerkungen:

- Andere Konstruktionen an Gitterverstärkungen sind möglich, auch mit rand-verstärkten Zonen.

D-Teningen, September 1988 - Kr/pw

11.51



Flexible Packstoffe - Technische Verbunde

**Tscheulin-Rothal GmbH**

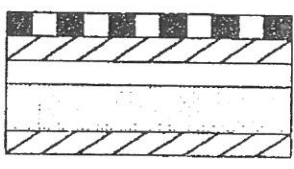
Ein Unternehmen der VAW Europack Gruppe

Bausolierungen  
Segment 81/18

<b>PATENTBESVÄRSRÄTTEN</b>	
Ink	2010 -08- 1.3
Mål nr	Aktbil
10-124	9

**Aluminium-Gelege-Aluminiumverbund gefärbt**

Materialaufbau:



- schwerentflammbare Deckweißfärbung
- Aluminiumfolie 0,020 mm weich
- Spezialkaschierung
- Glasfadengelege RG 2/2 34 PVA 5 x 5 mm beidseitig 80 mm Randverstärkung
- Aluminiumfolie 0,012 mm weich

Lieferform: Rollen

Eigenschaften:

- in Verbindung mit Mineralfaserdämmplatten verklebt schwer entflammbar B1 nach DIN 4102
- hochreißfest
- wasserdampfdicht

Verwendungszweck:

Bausolation, im speziellen Deckschicht für Mineral-, Glaswoll- bzw. Steinwoll-Matten bzw. -Platten

Bemerkungen:

- andere Glasfadengelegekonstruktionen sind möglich
- max. Rollenbreite ca. 1 360 mm

D-Teningen, August 1990 - Kr/pw



D12

# Polyethylene terephthalate

From Wikipedia, the free encyclopedia

PATENTBESVÄRSRÄTTEN	
Ink	2010 -08- 1.3
Mål nr	10-124
Aktbil	A 10
PET	

**Polyethylene terephthalate** (sometimes written poly(ethylene terephthalate)), commonly abbreviated **PET**, **PETE**, or the obsolete **PETP** or **PET-P**), is a thermoplastic polymer resin of the polyester family and is used in synthetic fibers; beverage, food and other liquid containers; thermoforming applications; and engineering resins often in combination with glass fiber.

Depending on its processing and thermal history, polyethylene terephthalate may exist both as an amorphous (transparent) and as a semi-crystalline material. The semi crystalline material might appear transparent (spherulites < 500 nm) or opaque and white (spherulites up to a size of some μm) depending on its crystal structure and spherulite size. Its monomer (bis-β-hydroxyterephthalate) can be synthesized by the esterification reaction between terephthalic acid and ethylene glycol with water as a byproduct, or by transesterification reaction between ethylene glycol and dimethyl terephthalate with methanol as a byproduct. Polymerization is through a polycondensation reaction of the monomers (done immediately after esterification/transesterification) with ethylene glycol as the byproduct (the ethylene glycol is directly recycled in production).

The majority of the world's PET production is for synthetic fibers (in excess of 60%) with bottle production accounting for around 30% of global demand. In discussing textile applications, PET is generally referred to as simply "polyester" while "PET" is used most often to refer to packaging applications.

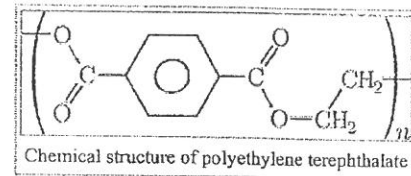
Some of the trade names of PET products are **Dacron**, **Diolen**, **Tergal**, **Terylene**, and **Trevira** fibers.<sup>[1]</sup> **Clartuf**, **Eastman PET** and **Polyclear** bottle resins, **Hostaphan**, **Melinex**, and **Mylar** films, and **Arnite**, **Ertalyte**, **Impet**, **Rynite** and **Valox** injection molding resins. The polyester industry makes up about 18% of world polymer production and is third after polyethylene (PE) and polypropylene (PP).

PET consists of polymerized units of the monomer ethylene terephthalate, with repeating C<sub>10</sub>H<sub>8</sub>O<sub>4</sub> units. It contains the chemical elements carbon, hydrogen, and oxygen. If fully burned, it produces only carbon dioxide (CO<sub>2</sub>) and water (H<sub>2</sub>O). PET is commonly recycled, and has the number "1" as its recycling symbol.

Molecular formula	(C <sub>10</sub> H <sub>8</sub> O <sub>4</sub> ) <sub>n</sub>
Density amorphous	1370 kg/m <sup>3</sup>
Density crystalline	1455 kg/m <sup>3</sup>
Young's modulus(E)	2800–3100 MPa
Tensile strength(σ <sub>t</sub> )	55–75 MPa
Elastic limit	50–150%
notch test	3.6 kJ/m <sup>2</sup>
Glass temperature	75 °C
melting point	260 °C
Vicat B	170 °C
Thermal conductivity	0.24 W/(m·K)
linear expansion coefficient (α)	7×10 <sup>-5</sup> /K
Specific heat (c)	1.0 kJ/(kg·K)
Water absorption (ASTM)	0.16
Refractive Index	1.5750
Price	0.5–1.25 €/kg
source: A.K. van der Vegt & L.E. Govaert, Polymeren, van keten tot kunstof, ISBN 90-407-2388-5	

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## Uses

PET can be semi-rigid to rigid, depending on its thickness, and is very lightweight. It makes a good gas and fair moisture barrier, as well as a good barrier to alcohol (requires additional "Barrier" treatment) and solvents. It is strong and impact-resistant. It is naturally colorless with high transparency.

PET bottles are excellent barrier materials and are widely used for soft drinks (see carbonation). For certain specialty bottles, PET sandwiches an additional polyvinyl alcohol to further reduce its oxygen permeability.

When produced as a thin film (biaxially oriented PET film, often known by one of its tradenames, "Mylar"), PET can be aluminized by evaporating a thin film of metal onto it to reduce its permeability, and to make it reflective and opaque (MPET). These properties are



useful in many applications, including flexible food packaging and thermal insulation, such as "space blankets". Because of its high mechanical strength, PET film is often used in tape applications, such as the carrier for magnetic tape or backing for pressure sensitive adhesive tapes.

Non-oriented PET sheet can be thermoformed to make packaging trays and blisters. If crystallizable PET is used, the trays can be used for frozen dinners, since they withstand both freezing and oven baking temperatures.

When filled with glass particles or fibers, it becomes significantly stiffer and more durable. This glass-filled plastic, in a semi-crystalline formulation, is sold under the tradename **Rynite**, **Arnite**, **Hostadur**, and **Crastin**.



While most thermoplastics can, in principle, be recycled, PET bottle recycling is more practical than many other plastic applications. The primary reason is that plastic carbonated soft drink bottles and water bottles are almost exclusively PET, which makes them more easily identifiable in a recycle stream. PET has a resin identification code of 1. One of the uses for a recycled PET bottle is for the manufacture of polar fleece material. It can also make fiber for polyester products.

Because of the recyclability of PET and the relative abundance of post-consumer waste in the form of bottles, PET is rapidly gaining market share as a carpet fiber. Leading the way, Mohawk Industries released everSTRAND in 1999, a 100% post-consumer recycled content PET fiber. Since that time, more than 17 billion bottles have been recycled into carpet fiber.<sup>[2]</sup> Pharr Yarns, a supplier to numerous carpet manufacturers including Looptex, Dobbs Mills, and Berkshire Flooring,<sup>[3]</sup> produces a BCF (bulk continuous filament) PET carpet fiber containing a minimum of 25% post-consumer recycled content.

PET, as with many plastics, is also an excellent candidate for thermal disposal (incineration), as it is composed of carbon, hydrogen, and oxygen, with only trace amounts of catalyst elements (but no sulphur). PET has the energy content of soft coal.

PET was patented in 1941 by the Calico Printers' Association of Manchester. The PET bottle was patented in 1973.

## Intrinsic viscosity

One of the most important characteristics of PET is referred to as I.V. (intrinsic viscosity).

The I.V. of the material, measured in deciliters per gram (dl/g) is dependent upon the length of its polymer chains. The longer the chains, the stiffer the material, and therefore the higher the I.V. The average chain length of a particular batch of resin can be controlled during polymerization.

An I.V. of about:

- 0.60 dl/g: Would be appropriate for fibre
- 0.65 dl/g: Film
- 0.76-0.84 dl/g: Bottles
- 0.85 dl/g: Tire cord

## Drying

PET is hygroscopic, meaning that it naturally absorbs water from its surroundings. However, when this 'damp' PET is then heated, the water hydrolyzes the PET, decreasing its resilience. This means that before the resin can be processed in a molding machine, as much moisture as possible must be removed from the resin. This is achieved through the use of a desiccant or dryers before the PET is fed into the processing equipment.

Inside the dryer, hot dry air is pumped into the bottom of the hopper containing the resin so that it flows up through the pellets, removing moisture on its way. The hot wet air leaves the top of the hopper and is first run through an after-cooler, because it is easier to remove moisture from cold air than hot air. The resulting cool wet air is then passed through a desiccant bed. Finally the cool dry air leaving the desiccant bed is re-heated in a process heater and sent back through the same processes in a closed loop. Typically residual moisture levels in the resin must be less than 5 parts per million (parts of water per million parts of resin, by weight) before processing. Dryer residence time should not be shorter than about four hours. This is because drying the material in less than 4 hours would require a temperature above 160 °C, at which level hydrolysis would begin inside the pellets before they could be dried out.

PET can also be dried in compressed air resin dryers. Compressed air dryers do not reuse drying air. Dry, heated compressed air is circulated through the PET pellets as in the desiccant dryer, then released to the atmosphere.

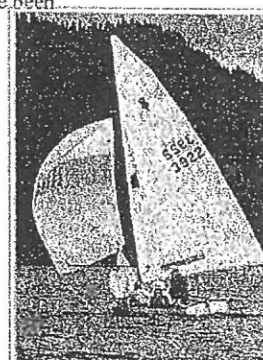
## Copolymers

In addition to pure (homopolymer) PET, PET modified by copolymerization is also available.

In some cases, the modified properties of copolymer are more desirable for a particular application. For example, cyclohexane dimethanol (CHDM) can be added to the polymer backbone in place of ethylene glycol. Since this building block is much larger (6 additional carbon atoms) than the ethylene glycol unit it replaces, it does not fit in with the neighbouring chains the way an ethylene glycol unit would. This interferes with crystallization and lowers the polymer's melting temperature. Such PET is generally known as PETG (Eastman Chemical and SK Chemicals are the only two manufacturers). PETG is a clear amorphous thermoplastic that can be injection molded or sheet extruded. It can be colored during processing.



A PET soft drink bottle



Sails are usually made of Dacron, a brand of PET fiber; colorful lightweight spinnakers are usually made of nylon.

Another common modifier is isophthalic acid, replacing some of the 1,4-(*para*-) linked terephthalate units. The 1,2-(*ortho*-) or 1,3-(*meta*-) linkage produces an angle in the chain, which also disturbs crystallinity.

Such copolymers are advantageous for certain molding applications, such as thermoforming, which is used for example to make tray or blister packagings from PETG film, or PETG sheet. On the other hand, crystallization is important in other applications where mechanical and dimensional stability are important, such as seat belts. For PET bottles, the use of small amounts of CHDM or other comonomers can be useful: if only small amounts of comonomers are used, crystallization is slowed but not prevented entirely. As a result, bottles are obtainable via stretch blow molding ("SBM"), which are both clear and crystalline enough to be an adequate barrier to aromas and even gases, such as carbon dioxide in carbonated beverages.

## Crystals

Crystallization occurs when polymer chains fold up on themselves in a repeating, symmetrical pattern. Long polymer chains tend to become entangled on themselves, which prevents full crystallization in all but the most carefully controlled circumstances. PET is no exception to this rule; 60% crystallization is the upper limit for commercial products, with the exception of polyester fibers.

PET in its natural state is a crystalline resin. Clear products can be produced by rapidly cooling molten polymer to form an amorphous solid. Like glass, amorphous PET forms when its molecules are not given enough time to arrange themselves in an orderly fashion as the melt is cooled. At room temperature the molecules are frozen in place, but if enough heat energy is put back into them, they begin to move again, allowing crystals to nucleate and grow. This procedure is known as solid-state crystallization.

Like most materials, PET tends to produce many small crystallites when crystallized from an amorphous solid, rather than forming one large single crystal. Light tends to scatter as it crosses the boundaries between crystallites and the amorphous regions between them. This scattering means that crystalline PET is opaque and white in most cases. Fiber drawing is among the few industrial processes that produces a nearly single-crystal product.

## Degradation

PET is subject to various types of degradations during processing. The main degradations that can occur are hydrolytic, thermal and probably most important thermal oxidation. When PET degrades, several things happen: discoloration, chain scissions resulting in reduced molecular weight, formation of acetaldehyde and cross-links ("gel" or "fish-eye" formation). Discoloration is due to the formation of various chromophoric systems following prolonged thermal treatment at elevated temperatures. This becomes a problem when the optical requirements of the polymer are very high, such as in packaging applications. Acetaldehyde is normally a colorless, volatile substance with a fruity smell. It forms naturally in fruit, but it can cause an off-taste in bottled water. Acetaldehyde forms in PET through the "abuse" of the material. High temperatures (PET decomposes above 300 °C or 570 °F), high pressures, extruder speeds (excessive shear flow raises temperature) and long barrel residence times all contribute to the production of acetaldehyde. When acetaldehyde is produced, some of it remains dissolved in the walls of a container and then diffuses into the product stored inside, altering the taste and aroma. This is not such a problem for non-consumables (such as shampoo), for fruit juices (which already contain acetaldehyde), or for strong-tasting drinks like soft drinks. For bottled water, however, low acetaldehyde content is quite important, because if nothing masks the aroma, even extremely low concentrations (10-20 parts per billion in the water) of acetaldehyde can produce an off-taste. The thermal and thermooxidative degradation results in poor processibility characteristics and performance of the material.

One way to alleviate this is to use a copolymer. Comonomers such as CHDM or isophthalic acid lower the melting temperature and reduces the degree of crystallinity of PET (especially important when the material is used for bottle manufacturing). Thus the resin can be plastically formed at lower temperatures and/or with lower force. This helps to prevent degradation, reducing the acetaldehyde content of the finished product to an acceptable (that is, unnoticeable) level. See copolymers, above.

Other ways to improve the stability of the polymer is by using stabilizers, mainly antioxidants such as phosphites. Recently, molecular level stabilization of the material using nanostructured chemicals has also been considered.

## Antimony

Antimony (Sb) is a catalyst that is often used as Antimony trioxide (Sb<sub>2</sub>O<sub>3</sub>) or Antimony triacetate in the production of PET. It remains in the material and can thus in principle migrate out into food and drinks. Although antimony trioxide is of low toxicity, its presence is still of concern. The Swiss Federal Office of Public Health investigated the amount of antimony migration, comparing waters bottled in PET and glass: the antimony concentrations of the water in PET bottles was higher, but still well below the allowed maximal concentrations.<sup>[4]</sup> (report available in German and French only) The Swiss Federal Office of Public Health concluded that small amounts of antimony migrate from the PET into bottled water, but that the health risk of the resulting low concentrations is negligible (1% of the "tolerable daily intake" determined by the WHO). A later (2006) but more widely publicized study by a group of geochemists at the University of Heidelberg headed by William Shotyk found similar amounts of antimony in water in PET bottles.<sup>[5]</sup>

The WHO also has also published a risk assessment for antimony in drinking water<sup>[6]</sup>.

## Bottle processing equipment

There are two basic molding methods for PET bottles, one-step and two-step. In two-step molding, two separate machines are used. The first machine injection molds the preform. The preform looks like a test tube. The bottle-cap threads are already molded into place, and the body of the tube is significantly thicker, as it will be inflated into its final shape in the second step using stretch blow molding.

In the second process, the preforms are heated rapidly and then inflated against a two-part mold to form them into the final shape of the

bottle. Preforms (uninflated bottles) are now also used as containers for candy.

In one-step machines, the entire process from raw material to finished container is conducted within one machine, making it especially suitable for molding non-standard shapes (custom molding), including jars, flat oval, flask shapes etc. Its greatest merit is the reduction in space, product handling and energy, and far higher visual quality than can be achieved by the two-step system.

## Polyester recycling industry

### Introduction

When recycling polyethylene terephthalate or PET or polyester, two ways generally have to be differentiated:

A: The chemical recycling back to the initial raw materials purified terephthalic acid (PTA) or dimethyl terephthalate (DMT) and monoethylene glycol (MEG) where the polymer structure is destroyed completely, or in process intermediates like bis-β-hydroxyterephthalate

B: The mechanical recycling where the original polymer properties are being maintained or reconstituted.

A: Chemical recycling of PET will become cost-efficient only applying high capacity recycling lines of >> 50,000 tons/year. Such lines could only be seen, if at all, within the production sites of very large polyester producers. Several attempts of industrial magnitude to establish such chemical recycling plants are made in the past but without resounding success till 2008. Even the promising chemical recycling in Japan became not an industrial breakthrough so far. The two reasons for this are at first the difficulty of consistent and continuous waste bottles sourcing in such a huge amount at one single site and at second the steadily increased prices and price volatility of collected bottles. The prices of baled bottles increased for instance between the years 2000 and 2008 from about 50 Euro/ton to over 500 Euro/ton in 2008.

Mechanical recycling or direct circulation of PET in the polymeric state is operated in most diverse variants today. These kinds of processes are typical of small and medium-sized industry. Cost-efficiency can already be achieved with plant capacities within a range of 5 000 – 20 000 tons/year. In this case, nearly all kinds of recycled-material feedback into the material circulation are possible today. These diverse recycling processes are being discussed hereafter in detail.

Besides chemical contaminants and degradation products generated during first processing and usage, mechanical impurities are representing the main part of quality depreciating impurities in the recycling stream. Due to the trend that recycled materials are increasingly introduced into manufacturing processes, which were originally designed for new materials only, efficient sorting, separation and cleaning processes become most important for high quality recycled polyester.

When talking about polyester recycling industry we are concentrating mainly on recycling of PET bottles which are meanwhile used for all kinds of liquid packaging like water, carbonated soft drinks, juices, beer, sauces, detergents, household chemicals and so on. Bottles are easily to distinguish because of shape and consistency and separate from waste plastic streams either by automatic or hand sorting processes. The established polyester recycling industry exists of three major sections:

- PET bottle collection and waste separation—waste logistics
- Production of clean bottle flakes—flake production
- Conversion of PET flakes to final products—flake processing

Intermediate product from the first section is baled bottle waste with a PET content greater than 90%. Most common trading form is the bale but also bricked or even loose, pre-cut bottles are common in the market. In the second section the collected bottles are converted to clean PET bottle flakes. This step can be more or less complex and complicated depending on required final flake quality. During third step PET bottle flakes are processed to any kind of products like film, bottles, fiber, filament, strapping or intermediates like pellets for further processing and engineering plastics.

Aside this external polyester bottle recycling numbers of internal recycling processes exist, where the wasted polymer material does not exit the production site to the free market and where the waste is reused at one and the same production circuit. In this way for instance fiber waste is directly reused to produce fiber, preform waste is directly reused to produce preforms and film waste is directly reused to produce film.<sup>[7]</sup>

### PET bottle recycling

#### Purification and decontamination – the most important processing steps during polyester recycling

The success of any recycling concept is hidden in the efficiency of purification and decontamination at the right place during processing and to the necessary or desired extent.

Generally, the following applies: the sooner foreign substances are removed, in the process, and the more thoroughly this is done, the more efficient the process is.

The high plasticization temperature of PET in the range of 280°C is the reason why almost all common organic impurities such as PVC, PLA, polyolefin, chemical wood-pulp and paper fibers, polyvinyl acetate, melt adhesive, coloring agents, sugar and proteins residues are transformed into colored degradation products which, in their turn, might release reactive degradation products additionally. Then, the number of defects in the polymer chain increases considerably. Naturally, the particle size distribution of impurities is very wide, the big particles of 1 000 - > 60 µm - which are visible by naked eye and easy to filtrate - representing the lesser evil since their total surface is relatively small and the degradation speed is therefore lower. The influence of the microscopic particles, which – because they are many - increase the frequency of defects in the polymer, is comparable bigger.

The motto "What the eye does not see the heart cannot grieve over" is considered to be very important in many recycling processes.

Therefore besides efficient sorting the removal of visible impurity particles by melt filtration processes is playing a particular part in this case.

In general one can say that the processes to make PET bottle flakes from collected bottles are as versatile as the different waste streams are different in their composition and quality. In view of technology there isn't just one way to do it. There are meanwhile many engineering companies which are offering flake production plants and components and it is difficult to decide for one or other plant design. Nevertheless there are principles which are sharing most of these processes. Depending on composition and impurity level of input material the general following process steps are applied.<sup>[8]</sup>

1. Bale opening, briquette opening
2. Sorting and selection for different colors, foreign polymers especially PVC, foreign matter, removal of film, paper, glass, sand, soil, stones and metals
3. Pre-washing without cutting
4. Coarse cutting dry or combined to pre-washing
5. Removal of stones, glass and metal
6. Air sifting to remove film, paper and labels
7. Grinding, dry and / or wet
8. Removal of low density polymers (cups) by density differences
9. Hot wash
10. Caustic wash
11. Caustic surface etching, maintaining IV and decontamination
12. Rinsing
13. Clean water rinsing
14. Drying
15. Air sifting of flakes
16. Automatic flake sorting
17. Water circuit and water treatment technology
18. Flake quality control

#### Impurities and material defects

The number of possible impurities and material defects which accumulate in the polymeric material is increasing permanently - when processing as well as when using polymers - taking into account a growing service life time, growing final applications and repeated recycling. As far as recycled PET bottles are concerned, the defects mentioned can be sorted in the following groups:

- a) Reactive polyester OH- or COOH- end groups are transformed into dead / not reactive end groups, e.g. formation of vinyl ester end groups through dehydration or decarboxylation of terephthalate acid, reaction of the OH- or COOH- end groups with mono-functional degradation products like mono-carbonic acids or alcohols. Results are decreased reactivity during re-polycondensation or re-SSP and broadening the molecular weight distribution.
- b) The end group proportion shifts toward the direction of the COOH end groups built up through a thermal and oxidative degradation. Results are decrease in reactivity, increase in the acid autocatalytic decomposition during thermal treatment in presence of humidity.
- c) Number of poly-functional macromolecules increases. Accumulation of gels and long-chain branching defects.
- d) Number, concentration and variety of non polymer-identical organic and inorganic foreign substances are increasing. With every new thermal stress, the organic foreign substances will react by decomposition. This is causing the liberation of further degradation-supporting substances and coloring substances.
- e) Due to service life of products made of polyester in the presence of air (oxygen) and humidity, as well as supported by ultraviolet light, hydro peroxide groups build up at the polymer surface. During an ulterior treatment process hydro peroxides are a source of oxygen-radicals which are source of oxidative degradation. Destruction of hydro peroxides is to happen before the first thermal treatment or during plasticization and can be supported by suitable additives like antioxidants.

Taking in consideration the above mentioned chemical defects and impurities, there is ongoing a modification of the following polymer characteristics during each recycling cycle, which are detectable by chemical and physical laboratory analysis.

In particular:

- Increase of COOH end groups
- Increase of color number b
- Increase of haze (transparent products)
- Increase of oligomer content
- Reduction in filterability
- Increase of by-products content such as acetaldehyde, formaldehyde
- Increase of extractable foreign contaminants
- Decrease in color L



- Decrease of intrinsic viscosity (IV) or the dynamic viscosity
- Decrease of crystallization temperature and increase of crystallization speed
- Decrease of the mechanical properties like tensile strength, elongation at break or elasticity modulus
- Broadening of molecular weight distribution

The recycling of PET-bottles is meanwhile an industrial standard process which is offered by a wide variety of engineering companies. [9]

### Processing examples for recycled polyester

Recycling processes with polyester are almost as varied as the manufacturing processes based on primary pellets or melt. Depending on purity of the recycled materials polyester can be used today in most of the polyester manufacturing processes as blend with virgin polymer or increasingly as 100% recycled polymer. Some exceptions like BOPET-film of low thickness, special applications like optical film or yarns through FDY-spinning at > 6000 m/min or microfilaments and micro-fibers are produced from virgin polyester only.

#### a) Simple re-pelletizing of bottle flakes

This process consists in transforming bottle waste into flakes, by drying and crystallizing the flakes, by plasticizing and filtering, as well as by pelletizing. Product is an amorphous re-granulate of an IV in the range of 0.55 - 0.7 dl/g, depending on how complete pre-drying of PET flakes has been done.

Special feature are: acetaldehyde and oligomers are contained in the pellets at lower level; the viscosity is reduced somehow, the pellets are amorphous and have to be crystallized and dried before further processing.

Processing to: Non-woven, Staple fiber, Filaments, Carpet yarn, A-PET film for thermoforming, Packaging stripes, BOPET packaging film, Bottle resin by SSP, Engineering plastics, Addition to PET virgin production.

Choosing the re-pelletizing way means having an additional conversion process which is at the one side energy intensive, cost consuming and causes thermal destruction. At the other side the pelletizing step is providing the following advantages:

- Quality uniformization
- Processing flexibility increased
- Product selection and separation by quality
- Intermediate quality control
- Intensive melt filtration
- Modification by additives

#### b) Manufacture of PET-pellets for bottles (B-2-B) and A-PET

This process is, in principle, similar to the one described above; however, the pellets produced are directly (continuously or discontinuously) crystallized and then subjected to a solid state polycondensation (SSP) in a tumbling drier or a vertical tube reactor. During this processing step, the corresponding IV of 0.80 – 0,085 dl/g is rebuild again and, at the same time, the acetaldehyde content is reduced to < 1ppm.

The fact that some machine manufacturers and line builders in Europe and USA make efforts to offer independent recycling processes, e.g. the so called bottle-to-bottle (B-2-B) process, such as URRC or BÜHLER, aims at generally furnishing proof of the "existence" of the required extraction residues and of the removal of model contaminants according to FDA applying the so called challenge test, which is necessary for the application of the treated polyester in the food sector. Besides this process approval it is nevertheless necessary that any user of such processes has to constantly check the FDA-limits for the raw materials manufactured by himself for his process.

#### c) Direct conversion of bottle flakes

In order to save costs, one is working on the direct use of the PET-flakes, from the treatment of used bottles, with a view to manufacturing an increasing number of polyester intermediates. For the adjustment of the necessary IV, besides an efficient drying of the flakes, it is possibly necessary to also reconstitute the IV through polycondensation in the melt phase or solid state polycondensation of the flakes. The latest PET flake conversion processes are applying twin screw extruders, multi screw extruders or multi rotation systems and coincidental vacuum degassing to remove moisture and avoid flake pre-drying. These processes allow the conversion of undried PET flakes without substantial IV-drop caused by hydrolysis.

Looking at the consumption of PET bottle flakes the main portion of about 70% is converted to fibers and filaments. When using directly secondary materials such as bottle flakes in spinning processes, there are a few processing principles to obtain.

High speed spinning processes for the manufacture of POY normally need a spinning IV of 0.62 - 0.64 dl/g. Starting from bottle flakes, the IV can be set via the degree of drying. The additional use of TiO<sub>2</sub>-masterbatch is necessary for full dull or semi dull yarn. In order to protect the spinncrets, an efficient filtration of the melt is, in any case is necessary. For the time being the amount of POY made of 100% recycling polyester is rather low because this process requires high purity of spinning melt. Most of the time a blend of virgin and recycled pellets is used.

Staple fibers are spun in an IV-range which rather lies somewhat lower and which should be between 0.58 dl/g and 0.62 dl/g. In this

case, too, the required IV can be adjusted via drying or vacuum adjustment in case of vacuum extrusion. For adjusting the IV, however, an addition of chain length modifier like ethylene glycol or diethylene glycol can also be used.

Spinning non-woven - in the fine titer field for textile applications as well as heavy spinning non-woven as basic materials e.g. for roof covers or in road building - can be manufactured by spinning bottle flakes. The spinning IV is again within a range of 0.58 - 0,65 dl/g.

One field of increasing interest where recycled materials are used is the manufacture of high tenacity packaging stripes - and monofilaments. In both cases, the initial raw material is a mainly recycled material of higher IV. High tenacity packaging stripes as well as monofilament are then manufactured in the melt spinning process.

## Recycling back to the initial raw materials

### Glycolysis and partial glycolysis

The polyester which has to be recycled is transformed into an oligomer by adding ethylene glycol or other glycols during thermal treatment. The aim and advantage of this way of processing is the possibility of separating the mechanical deposits directly and efficient through a progressive and stepwise filtration. The filtration fineness of the last filtration step has a decisive effect on the quality of the end product. Taking partial recycling with partial glycolysis as an example, it is to be demonstrated how bottle waste can successfully be recycled in a continuously operating polyester line which is manufacturing pellets for bottle applications.

The task consists in feeding 10%- 25% bottle flakes and maintaining at the same time the quality of the bottle pellets which are manufactured on the line. This aim is solved by degrading the PET bottle flakes - already during their first plasticization which can be carried out in a single- or multi-screw extruder - to an IV of approx. 0.30 dl/g by adding small quantities of ethylene glycol and by subjecting the low viscosity melt stream to an efficient filtration directly after plasticization. Furthermore, temperature is brought to the lowest possible limit. In addition, with this way of processing, the possibility of a chemical decomposition of the hydro peroxides is possible by adding a corresponding P-stabilizer directly when plasticizing. The destruction of the hydro peroxide groups is, with other processes, already carried out during the last step of flake treatment for instance by adding  $H_3PO_3$ , see also.<sup>[10]</sup> The partially glycolysed and finely filtered recycled material is continuously fed to the esterification or prepolycondensation reactor, the dosing quantities of the raw materials are being adjusted accordingly.

The treatment of polyester waste through total glycolysis to convert the polyester to bis-beta hydroxy-terephthalate, which is vacuum distilled and can be used, instead of DMT or PTA, as a raw material for polyester manufacture, has been executed on an industrial scale in Japan as experimental production.

### Hydrolysis

Recycling processes, through hydrolysis of the PET to PTA and MEG, are operating under high pressures under supercritical conditions. In this case, PET-waste will be directly hydrolyzed applying for instance supercritical water steam. Purification of crude terephthalic acid will be carried out by re-crystallization in acetic acid / water mixtures similar to PTA purification. Industrial-scale lines based on this chemistry have not been known to date.

### Methanolysis

Methanolysis is the recycling process which has been practiced and tested on a large scale for many years in the past. In this case, polyester waste is transformed with methanol into DMT, under pressure and in presence of catalysts. After this an efficient filtration of the methanolysis product is applied. Finally the crude DMT is purified by vacuum distillation. The methanolysis is only rarely carried out in industry today because polyester production based on DMT shrunk tremendously and with this DMT producers disappeared step by step during the last decade. See also.<sup>[11]</sup>

## Notes and references

- <sup>1</sup> ^ Polyethylene terephthalate - Goodfellow, online source, sources, small quantity, quantities
- <sup>2</sup> ^ <http://www.mohawkflooring.com/carpeting/everstrand/default.aspx> Mohawk Flooring - everStrand
- <sup>3</sup> ^ <http://www.simplygreencarpet.com/> Simply Green Carpet - A Berkshire Flooring Brand
- <sup>4</sup> ^ <http://www.bag.admin.ch/dokumentation/publikationen/01435/01796/index.html?lang=de&download=M3wBPgDB/8ull6Dn36WcnojN14in3qSbnpWWZWjXmE6p1rJgsYfhyt3NhqbdqIV+baqwbKbXrZ6lluDZz8mMps2go6fo>
- <sup>5</sup> ^ [http://www.isc.org/delivery/\\_ArticleLinking/DisplayArticleForFree.cfm?doi=b517844b&JournalCode=EM](http://www.isc.org/delivery/_ArticleLinking/DisplayArticleForFree.cfm?doi=b517844b&JournalCode=EM)
- <sup>6</sup> ^ [http://www.who.int/water\\_sanitation\\_health/dwq/chemicals/antimonysum.pdf](http://www.who.int/water_sanitation_health/dwq/chemicals/antimonysum.pdf)
- <sup>7</sup> ^ <http://www.polyester-technology.com/index.html> - Collection of Publications - "Recycling Concepts for Polyester"
- <sup>8</sup> ^ "Current Technological Trends in Polyester Recycling", ISBN 3-00-019765-6; PETplanet Publisher GmbH, Heidelberg, Germany ISBN 978-3-9807497-4 ; 9th International Polyester Recycling Forum Washington / Sao Paulo
- <sup>9</sup> ^ "Polyester Bottle Resins Production, Processing, Properties and Recycling" by Dr. Ulrich K. Thiele; PETplanet Publisher GmbH, Heidelberg, Germany ISBN 978-3-9807497-4-9; chapter H, 2007
- <sup>10</sup> ^ DE-Patent DE19503055
- <sup>11</sup> ^ Stoyko Fakirov; „Handbook of Polyester“, ISBN 3-527-30113-5 – Wiley-VCH, Weinheim, 2002, pages 1223 ff

## See also

- Recycling of PET Bottles
- Biaxially oriented PET film
- Plastic recycling
- Polyester
- Solar water disinfection - a method of disinfecting water using only sunlight and plastic PET bottles

## External links

- American Plastics Council: PlasticInfo.org
- KenPlas Industry Ltd.: "What is PET (Polyethylene Terephthalate)"

Retrieved from "[http://en.wikipedia.org/wiki/Polyethylene\\_terephthalate](http://en.wikipedia.org/wiki/Polyethylene_terephthalate)"

Categories: Aromatic compounds | Carboxylate esters | Plastics | Household chemicals | Polyesters | Recyclable materials | Polymers | Thermoplastics | Transparent materials

Hidden categories: Articles needing additional references from May 2008 | All articles with unsourced statements | Articles with unsourced statements from March 2009

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## Standard Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus)<sup>1</sup>

This standard is issued under the fixed designation D 36; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

### 1. Scope

1.1 This test method covers the determination of the softening point of bitumen in the range from 30 to 157°C (86 to 315°F) using the ring-and-ball apparatus immersed in distilled water (30 to 80°C), USP glycerin (above 80 to 157°C), or ethylene glycol (30 to 110°C).

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

- 2.1 *ASTM Standards:*
- C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials<sup>2</sup>
  - D 92 Test Method for Flash and Fire Points by Cleveland Open Cup<sup>3</sup>
  - D 140 Practice for Sampling Bituminous Materials<sup>4</sup>
  - D 3461 Test Method for Softening Point of Asphalt and Pitch (Mettler Cup-and-Ball Method)<sup>5</sup>
  - E 1 Specification for ASTM Thermometers<sup>6</sup>

### 3. Summary of Test Method

3.1 Two horizontal disks of bitumen, cast in shouldered brass rings, are heated at a controlled rate in a liquid bath while each supports a steel ball. The softening point is reported as the mean of the temperatures at which the two disks soften enough to allow each ball, enveloped in bitumen, to fall a distance of 25 mm (1.0 in.).

### 4. Significance and Use

4.1 Bitumens are viscoelastic materials without sharply defined melting points; they gradually become softer and less

viscous as the temperature rises. For this reason, softening points must be determined by an arbitrary and closely defined method if results are to be reproducible.

4.2 The softening point is useful in the classification of bitumens, as one element in establishing the uniformity of shipments or sources of supply, and is indicative of the tendency of the material to flow at elevated temperatures encountered in service.

### 5. Apparatus

5.1 *Rings*—Two square-shouldered brass rings conforming to the dimensions shown in Fig. 1(a).

5.2 *Pouring Plate*—A flat, smooth, brass plate approximately 50 by 75 mm (2 by 3 in.).

5.3 *Balls*—Two steel balls, 9.5 mm (3/8 in.) in diameter, each having a mass of 3.50 ± 0.05 g.

5.4 *Ball-Centering Guides*—Two brass guides for centering the steel balls, one for each ring, conforming to the general shape and dimensions shown in Fig. 1 (b).

5.5 *Bath*—A glass vessel, capable of being heated, not less than 85 mm in inside diameter and not less than 120 mm in depth from the bottom of the flare.

NOTE 1—An 800-mL, low-form Griffin beaker of heat-resistant glass meets this requirement.

5.6 *Ring Holder and Assembly*—A brass holder designed to support the two rings in a horizontal position, conforming to the shape and dimensions shown in Fig. 1 (c), supported in the assembly illustrated in Fig. 1 (d). The bottom of the shouldered rings in the ring holder shall be 25 mm (1.0 in.) above the upper surface of the bottom plate, and the lower surface of the bottom plate shall be 16 ± 3 mm (5/8 ± 1/8 in.) from the bottom of the bath.

#### 5.7 Thermometers:

5.7.1 An ASTM Low Softening Point Thermometer, having a range from -2 to +80°C or 30 to 180°F, and conforming to the requirements for Thermometer 15C or 15F as prescribed in Specification E 1.

5.7.2 An ASTM High Softening Point Thermometer, having a range from 30 to 200°C or 85 to 392°F, and conforming to the requirements for Thermometer 16C or 16F as prescribed in Specification E 1.

5.7.3 The appropriate thermometer shall be suspended in the assembly as shown in Fig. 1 (d) so that the bottom of the bulb is level with the bottom of the rings and within 13 mm (0.5 in.) of the rings, but not touching them or the ring holder.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-8 on Roofing, Waterproofing, and Bituminous Materials and is the direct responsibility of Subcommittee D08.03 on Surfacing and Bituminous Materials for Membrane Waterproofing and Builtup Roofing.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 04.02.

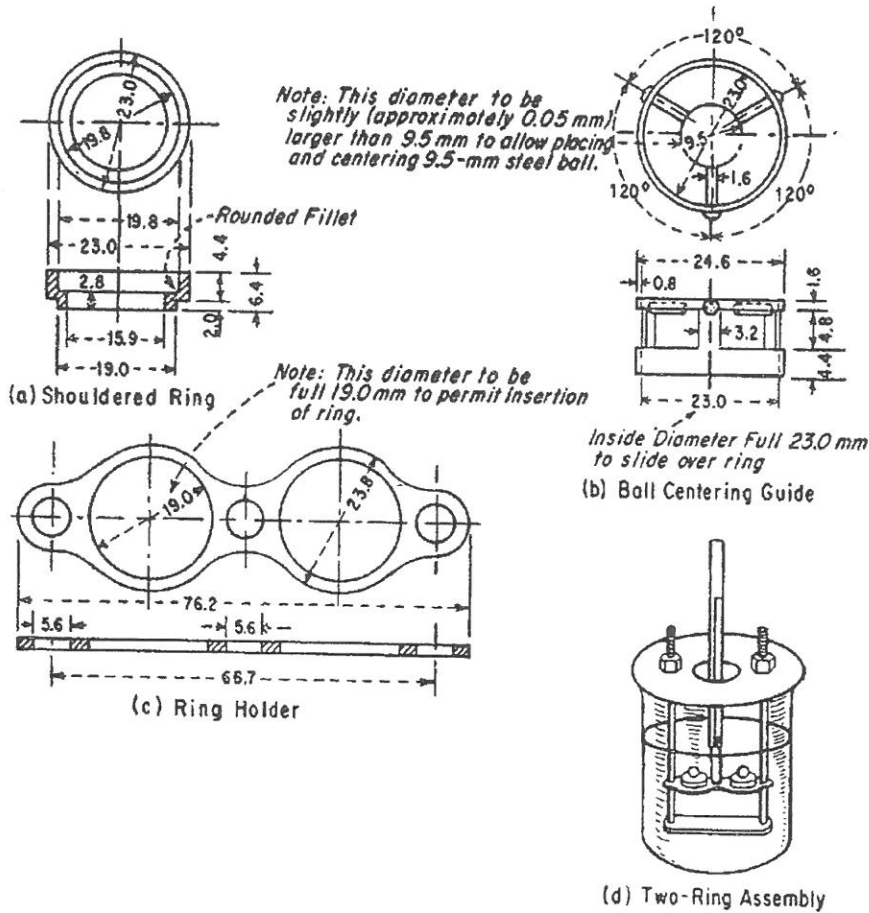
<sup>3</sup> Annual Book of ASTM Standards, Vol 05.01.

<sup>4</sup> Annual Book of ASTM Standards, Vol 04.03.

<sup>5</sup> Annual Book of ASTM Standards, Vol 04.04.

<sup>6</sup> Annual Book of ASTM Standards, Vol 14.03.





NOTE 1—All dimensions are in millimetres.  
 FIG. 1 Shouldered Ring, Ball-Centering Guide, Ring Holder, and Assembly of Apparatus Showing Two Rings

Substitution of other thermometers shall not be permitted.

**6. Reagents and Materials**

**6.1 Bath Liquids:**

**6.1.1 Freshly Boiled Distilled Water.**

NOTE 2—The use of freshly boiled distilled water is essential to avoid trapping air bubbles on the surface of the specimen which may affect the results.

**6.1.2 USP Glycerin, or**

NOTE 3—Caution: Glycerin has a flash point of 160°C (320°F) in accordance with Test Method D 92.

**6.1.3 Ethylene Glycol, with a boiling point between 195 and 197°C (383 and 387°F).**

NOTE 4—Caution: Ethylene glycol is toxic when taken internally or inhaled as a vapor. Avoid prolonged or repeated skin contact and inhalation of vapors. Its flash point is 115°C (239°F) in accordance with Test Method D 92. When using this bath liquid, conduct the test in a vented laboratory hood with adequate exhaust fan capacity to ensure removal of toxic vapors.

**6.2 Release Agents:**

6.2.1 To prevent adhesion of bitumen to the pouring plate when casting disks, the surface of the brass pouring plate may be thinly coated just before use with silicone oil or grease (Note 5), a mixture of glycerin and dextrin, talc, or china clay.

NOTE 5—Caution: Isolate silicones from other bituminous testing equipment and samples to avoid contamination, and wear disposable rubber gloves whenever handling silicones or apparatus coated with them. Silicone contamination can produce erroneous results in other tests such as those for penetration and flash point.

**7. Sampling**

7.1 Sample the material in accordance with Practice D 140.

**8. Test Specimens**

8.1 Do not start unless it is planned to complete preparation and testing of all asphalt specimens within 6 h and all coal-tar pitch specimens within 4½ h. Heat the bitumen sample with care, stirring frequently to prevent local overheating, until it has become sufficiently fluid to pour (Note 6). Stir carefully to avoid incorporation of air bubbles in the sample.

Note 6—An electric hot plate having a minimum power to unit-surface-area ratio of 37 kW/m<sup>2</sup> has been found satisfactory for this purpose.

8.1.1 Take no more than 2 h to heat an asphalt sample to its pouring temperature; in no case shall this be more than 110°C (200°F) above the expected softening point of the asphalt.

8.1.2 Take no more than 30 min to heat a coal-tar pitch sample to its pouring temperature; in no case shall this be more than 55°C (100°F) above the expected softening point of the coal-tar-pitch.

8.1.3 If the test must be repeated later, do not reheat this sample; use a fresh sample in a clean container to prepare new test specimens.

8.2 Heat the two brass rings (but not the pouring plate) to the approximate pouring temperature, and place them on the pouring plate treated with one of the release agents.

8.3 Pour a slight excess of the heated bitumen into each ring, and then allow the specimens to cool in ambient air for at least 30 min. For materials that are soft at room temperature, cool the specimens for at least 30 min at an air temperature at least 10°C (18°F) below the expected softening point. From the time the specimen disks are poured, no more than 240 min shall elapse before completion of the test.

8.4 When the specimens have cooled, cut away the excess bitumen cleanly with a slightly heated knife or spatula, so that each disk is flush and level with the top of its ring.

## 9. Procedure

9.1 Select one of the following bath liquids and thermometers appropriate for the expected softening point:

9.1.1 Freshly boiled distilled water for softening points between 30 and 80°C (86 and 176°F); use Thermometer 15C or 15F. The starting bath temperature shall be 5 ± 1°C (41 ± 2°F).

9.1.2 USP glycerin for softening points above 80°C (176°F) and up to 157°C (315°F); use Thermometer 16C or 16F. The starting bath temperature shall be 30 ± 1°C (86 ± 2°F).

9.1.3 Ethylene glycol for softening points between 30 and 110°C (86 and 230°F); use Thermometer 16C or 16F. The starting bath temperature shall be 5 ± 1°C (41 ± 2°F).

9.1.4 For referee purposes, all softening points up to 80°C (176°F) shall be determined in a water bath and all softening points above 80°C (176°F) shall be determined in a glycerin bath.

9.2 Assemble the apparatus in the laboratory hood with the specimen rings, ball-centering guides, and thermometer in position, and fill the bath so that the liquid depth will be 105 ± 3 mm (4 1/8 ± 1/8 in.) with the apparatus in place. If using ethylene glycol, make sure the hood exhaust fan is turned on and operating properly to remove toxic vapors. Using forceps, place the two steel balls in the bottom of the bath so they will reach the same starting temperature as the rest of the assembly.

9.3 Place the bath in ice water, if necessary, or gently heat to establish and maintain the proper starting bath temperature for 15 min with the apparatus in place. Take care not to contaminate the bath liquid.

9.4 Again using forceps, place a ball from the bottom of the bath in each ball-centering guide.

9.5 Heat the bath from below so that the temperature indicated by the thermometer rises at a uniform rate of 5°C

(9°F)/min (Note 7). Protect the bath from drafts, using shields if necessary. Do not average the rate of temperature rise over the test period. The maximum permissible variation for any 1-min period after the first 3 min shall be ± 0.5°C (± 1.0°F). Reject any test in which the rate of temperature rise does not fall within these limits.

Note 7—Rigid adherence to the prescribed heating rate is essential to reproducibility of results. Either a gas burner or electric heater may be used, but the latter must be of the low-lag, variable output type to maintain the prescribed rate of heating.

9.6 Record for each ring and ball the temperature indicated by the thermometer at the instant the bitumen surrounding the ball touches the bottom plate. Make no correction for the emergent stem of the thermometer. If the difference between the two temperatures exceeds 1°C (2°F), repeat the test.

## 10. Calculation

10.1 For a given bitumen specimen, the softening point determined in a water bath will be lower than that determined in a glycerin bath. Since the softening point determination is necessarily arbitrary, this difference matters only for softening points slightly above 80°C (176°F).

10.2 The change from water to glycerin for softening points above 80°C creates a discontinuity. With rounding, the lowest possible asphalt softening point reported in glycerin is 84.5°C (184°F), and the lowest possible coal-tar pitch softening point reported in glycerin is 82.0°C (180°F). Softening points in glycerin lower than these translate to softening points in water of 80°C (176°F) or less, and shall be so reported.

10.2.1 The correction for asphalt is -4.2°C (-7.6°F), and for coal-tar pitch is -1.7°C (-3.0°F). For referee purposes, repeat the test in a water bath.

10.2.2 Under any circumstances, if the mean of the two temperatures determined in glycerin is 80.0°C (176.0°F) or lower for asphalt, or 77.5°C (171.5°F) or lower for coal-tar pitch, repeat the test in a water bath.

10.3 To convert softening points slightly above 80°C (176°F) determined in water to those determined in glycerin, the correction for asphalt is +4.2°C (+7.6°F) and for coal-tar pitch is +1.7°C (+3.0°F). For referee purposes, repeat the test in a glycerin bath.

10.3.1 Under any circumstances, if the mean of the two temperatures determined in water is 85.0°C (185.0°F) or higher, repeat the test in a glycerin bath.

10.4 Results obtained by using an ethylene glycol bath will vary from those using water and glycerin. The following formulas shall be used to calculate the differences:

*Asphalt:*

$$SP(\text{glycerin}) = 1.026583 \times SP(\text{ethylene glycol}) - 1.334968^\circ\text{C} \quad (1)$$

$$SP(\text{water}) = 0.974118 \times SP(\text{ethylene glycol}) - 1.44459^\circ\text{C} \quad (2)$$

*Coal Tar:*

$$SP(\text{glycerin}) = 1.044795 \times SP(\text{ethylene glycol}) - 5.063574^\circ\text{C} \quad (3)$$

$$\dagger SP(\text{water}) = 1.061111 \times SP(\text{ethylene glycol}) - 8.413488^\circ\text{C} \quad (4)$$

† Editorially corrected.

## 11. Report

11.1 When using ASTM Thermometer 15C or 15F, report to the nearest 0.2°C or 0.5°F the mean or corrected mean of the temperatures recorded in 9.6 as the softening point.

11.2 When using ASTM Thermometer 16C or 16F report to the nearest 0.5°C or 1.0°F the mean or corrected mean of the temperatures recorded in 9.6 as the softening point.

11.3 Report the bath liquid used in the test.

## 12. Precision and Bias

12.1 With distilled water or USP glycerin, the following criteria shall be used for judging the acceptability of results (95 % probability):

12.1.1 *Single-Operator Precision*—The single-operator standard deviation has been found to be 0.41°C (0.73°F). Therefore, results of two properly conducted tests by the same operator on the same sample of bitumen should not differ by more than 1.2°C (2.0°F).<sup>7</sup>

12.1.2 *Multilaboratory Precision*—The multilaboratory standard deviation has been found to be 0.70°C (1.26°F).

Therefore, results of two properly conducted tests on the same sample of bitumen from two laboratories should not differ by more than 2.0°C (3.5°F).<sup>7</sup>

12.2 With ethylene glycol, the following criteria shall be used for judging the acceptability of results:

12.2.1 *Single-Operator Precision*—The single-operator standard deviation has been found to be 0.72°C (1.29°F). Therefore, results of two properly conducted tests by the same operator on the same sample of bitumen should not differ by more than 2.0°C (3.5°F).<sup>7</sup>

12.2.2 *Multilaboratory Precision*—The multilaboratory standard deviation has been found to be 1.08°C (1.95°F). Therefore, results of two properly conducted tests on the same sample of bitumen from two laboratories should not differ by more than 3.0°C (5.5°F).<sup>7</sup>

12.3 *Bias*—The procedure in Test Method D 36 has no bias because the value of the softening point of the bitumen test is defined in terms of this test method.

## 13. Keywords

13.1 asphalt; ball and ring; bitumen; coal tar; softening point

<sup>7</sup> These numbers represent, respectively, the (1S) and (D2S) limits as described in Practice C 670.

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