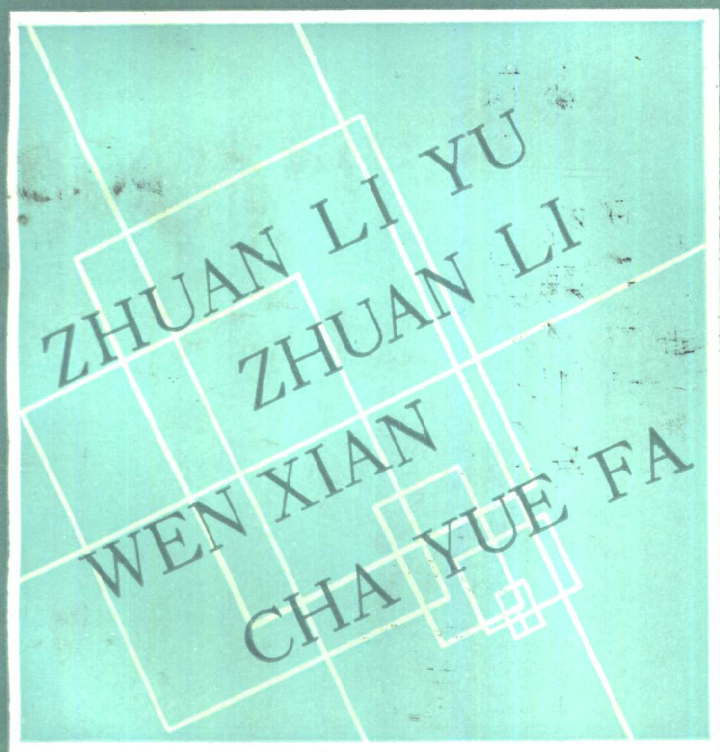


[美] J. T. 梅纳德 著

专利与专利文献 查阅法



化学工业出版社

专利与专利文献查阅法

〔美〕J. T. 梅纳德著

张玉华等译

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译 者 的 话

这本书好就好在写得具体。它回答了许多有关专利的具体问题。

譬如，为了能取得专利保护，在进行科研工作时该注意些什么？又譬如，根据什么来考虑并决定要不要申请专利？当决定申请专利了，又该怎么办理？还有，怎样才算侵犯别人的专利权？专利文献该怎么查找、怎么阅读？如何对付已经出现的专利纠纷？等等。这些问题，如果要从美国或其它国家的专利法中找答案，那将是些极为繁琐难解的法律条文；可是本书作者用通俗的文笔就事论事，却使我们读之易懂。

在我们国家，“专利”这个词汇已经越来越多地被某些领域的人们所谈起。当然，有的同志知之不详，只晓得它是个新玩意儿，自己想学学；有的则了解得多些，听说过实行专利制度会有利于促进科学技术和社会经济的发展；不少科技人员常查阅专利文献，从中获得有关世界各国新发明的情报；从事外贸工作的同志在与外国人打交道过程中也常涉及到专利问题；还有，我们有不少科研成果要报发明，还可能想有朝一日向外国申请专利…。总之，“专利”已经闯进我们的科研活动、经济生活和外贸工作中来了，不少同志脑际常会浮现出有关专利的种种问题，需要解答。为此，我们将本书译出（个别地方作了删节），希望能有助于我们进一步了解专利。另外，为便于广大读者利用专利文献，在文后补

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编了国际专利分类法简介、美国专利的检索方法、日本专利的检索方法、英国德温特出版公司检索刊物介绍，以供查找参考。

遗憾的是我们水平有限，不当之处敬请读者指出。另外，由于各国专利法不断在修改，书中会有已成或将成往迹的叙述，但无碍大局，只请读者注意。

译者谨志

序

本书是为化学家和化学工程师编写的。但是许多内容对于在其它领域工作而又需要了解美国专利制度的人也很有用。不过，本书的目的还是为了要回答那些在化学领域积极工作的人所经常涉及到的有关专利的问题。这本书是作者1976年春为杜邦公司实验站技术人员短训班讲课的一个产物。历来为这部分人所开设的各种课程中，听专利课的人数是最多的，这表明搞化工生产的人员对专利有着广泛的兴趣，也表明他们确实需要更多地了解专利，而听课人员所提的问题又在很大的程度上有助于作者确定该把哪些内容作为本书的重点最为相宜。

本书并不是关于专利法的书，也并非介绍专利许可证或办理过程的书，这方面的问题已有许多其它著作作了充分的叙述，其中最新近出版的，可能也是最好的一本，就是美国专利和商标局的 Peter Rosenberg 所写的《专利法基础知识》。本书没有引证关于专利案例的法院判决，对专利法细节也不想详加解释。因为这些只是专利专职人员的日常事务，对于技术人员来说并不太重要。

本书力求在以下几个方面回答化学家和工程师一些急切的实际问题：如何阅读与了解专利，如何利用专利做为一种情报源，如何确认一件发明已经完成，如何与律师或代理人一起工作以寻求用专利来保护发明、如何保存必要的笔记记录以及如何提防专利侵权等。本书关于如何实施和执行一项

专利讨论得很有限，这对从事实际工作的化学家来说关系不大。我们把法律和商务问题留给这方面的专业人员去考虑。不过，本书初稿曾由专门从事专利法律及商务方面的专家们审阅过，我们深信书中的观点是正确的。

专利这个领域也像别的专业一样有它自己专用的行话，本书的目的之一就是让技术人员能充分地熟悉这些行话，以便能自如地和专利律师、代理人及技术联络人员交往。对于在专利中专用的以及用于专利事务时有其特殊含义的字眼及用词，当它们在本书中首次出现时，多数情况下都用引号标出。书后还附有一个专利特定用词的词汇表，其中包括了在专利领域中常见的缩写词。

在实验站训练班执教的有杜邦公司法律部的专利律师 C. Harold Herr 和 Don M. Kerr，还有三位有经验的专利联络人：中心研究开发部的 Richard C. Toole，摄影产品部的 Frank H. Thompson 及化学弹性材料部的本书作者。在此，我要对这个教学小组出色的工作关系以及每位成员良好的讲授艺术谨致谢意。

我还要感谢美国化学协会的联合委员会——专利事务及有关立法委员会对编写本书所给予的鼓励，专利出版附属委员会成员及其主席 Willard Marcy 读过本书的初稿，他们的建议及意见是很有价值的，而 Florence Wood 出色的技巧和非凡的见识使本书初稿的写作成为一件快事而不至于象想像中的那样枯燥无味。

John T. Maynard
Wilmington, Del. 1977年4月

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第一章 引言：专利的意义

人类社会的技术进步，一直伴随着竞争活动，而现代人类则把竞争推向其最高形式；同时我们也懂得，为了使生产技术能更顺利地发展，我们就应当用一种合作的力量来和竞争的力量相平衡。哪个社会竞争与合作的平衡越有效，那么这个社会的技术进步也就越迅速。

专利制度就是如此一个成功的合作安排以及显示社会最佳作用的一个最直接的例子。

专利制度对于发明人所做的，具有竞争能力的创造性发明，给予暂时的、有限的独占权，以兹奖励，但同时也要求发明人与社会合作，即在此后的一段时期内，负责将其发明内容传授给社会的其他成员，使他们学会如何使用该项发明。如果一个人要有效地使用并且要了解专利，就必须牢记这个哲理，这将自始至终在本书中提到。

早期的专利制度

据人们所知，自从文艺复兴以来，专利制度就已被用作作为一个社会工具了。意大利城邦的统治者认识到，他的有创造性的臣民需要得到鼓励和保护，以免竞争对手抄袭。授给的专利权有不同的期限，最有名的一个就是由威尼斯的总督授给伽里略的为期20年的专利权，即关于灌溉农田的抽水机械的发明。欧洲及英国君主们用批准专利来鼓励贸易，但不幸的是他们也用此来酬劳宫庭宠臣。这后一情况的滥用，把独占权授给或卖给私人去从事特种商品贸易或事业，而完

全忽视了其创造性，结果导致英国议会于1624年通过了垄断法（The Statute of Monopoly）。这个法律规定专利权仅批准给对社会有真正贡献的人。一般来说，这部垄断法被认为是世界各国现有专利制度的基础。

美国的专利制度

美国远在殖民地时代就曾对发明人批准过专利，美国宪法制定者在宪法第一条第八款就给专利和版权法打下了基础。其条文内容是：

国会应当有权……为促进科学和有用技术的发展，确保作者和发明人在有限期间内对其作品和发明享有独占权。

1790年的专利法使宪法的这一条得以贯彻，它规定由国务卿Jefferson、军政部长Knox和检察长Randolph组成的委员会负责审查专利申请案。由George Washington, Randolph和Jefferson签署的第一件美国专利实际上是一件化学方面的发明。发明人是Samuel Hopkins，他对社会的贡献是改进了从草木灰制钾碱的方法，进而用于生产肥皂。

这个高级的委员会尽自己承担的义务，努力审查所有的申请是否具备专利价值，但是在不到三年的时间就明显地看到，委员会根本不能对提交的每件申请都给予充分的研究。1793年美国采用了“登记”制度，对每件符合格式的申请都予颁布专利，而将那些有冲突的专利申请的判别交给法院处理。

1936年，专利局发生一次大火，把这段混乱时期的全部记录统统付之一炬这可谓塞翁失马。接着国会于1836年通过了专利法，据此设置了专利局长的职位并成立了专利局，配备了足够的成员，负责审查发明申请案是否是真正的新发明。现在的美国专利就是采用这个专利法后重新按顺序编号

的，到1976年12月28日为止，已编到四百万号，第四百万号也是一件化学专利，是批给内华达州拉斯维加斯的 Robert Menden hall 的，发明内容为循环沥青-集料组分的方法。

1887年美国和其他许多国家加入了巴黎公约，公约规定每个缔约国的发明人享有一定范围的优先权。现行的美国专利法是1952年通过的专利法，其最为重要的一个新特点是为发明规定了一个切实可行的定义，揭示出必须具备哪些要素方能构成一件发明。编写本书时美国专利法正准备修订，对此将在第十三章中予以讨论。

当代其他类型的专利制度

多数国家目前都确立了专利制度。一些主要大国均采用“审查”制度——在批准专利前，要先将申请案与现有技术水平进行比较。较小的国家一般采用登记制度，即如果申请符合一定的格式要求，就自动地被批准为专利，专利权人的权利的实施则由法院处理。另外，许多小国往往是根据某一个大国先已颁布的专利而批准那些已经确定的专利。

专利仅在批准该件专利的国家里有效。因此发明人（或者是发明人的雇主）必须向打算求得专利保护的每一个国家提出专利申请。如果预期发明可有广泛的应用，这就将是一项花费很大的事情，所以常常有人希望一件发明只提出一次专利申请就可以取得世界范围的保护。这个愿望总有一天会实现，但肯定不会太快。

事实上，目前已有多种形式的专利合作行动正处于规划和通过的阶段。比如现在已通过的专利合作条约（PCT）规定，经过一次申请，并仅由一个审查组审查，就可获得所有签字国的认可。一次申请也可得到组成马尔加什联盟的17个法语非洲国家的认可。四个斯堪的纳维亚国家也有逐渐慢慢

走向共同专利的趋势。尽管每个国家还需根据申请人的要求和各国的专利法来颁布各自的专利，但是许多欧洲国家也在接受一次申请的做法（即欧洲专利制度）。所谓共同市场专利，不久也可在欧洲经济共同体的所有国家中办理一次申请。这些发展将在第十三章中详加讨论。

专利制度的性质和目的

世界上有许多专利制度，尽管它们在实施细节上千差万别，但是基本目标都是一致的，这就是鼓励每个发明人为了社会的长远利益公开其发明，而并不为得利而将其发明保密。这种鼓励就是给予发明人以短期的独占权利（期限长短各国不同，一般为5~20年），以防止（专利技术术语为“排除”）他人使用该项发明。

如果发明人履行了其义务，而这件发明又具备专利性要求的三个基本要素，则发明就被批准为专利。这三个要素^①是：

（1）确实是新的，技术术语是“新颖性”。

（2）不能根据已有知识显而易见。技术术语为“创造性”和“先进性”。

（3）发明必须是有用的。

每个国家的法律都按本国的方式来实施这些要求，即通过行政的或司法的程序来进行。但三个基本要素对所有专利制度都是共同的。

如果三个条件都符合了，就可能批准为专利。请特别注意“批准”这个词。专利是政府对发明人因公开了如何实施

① 我国通常将专利性的这三个要素概括为专利的“三性”，即新颖性，创造性和实用性。——校者

其发明而授予的酬报。这笔交易在双方之间成交，如果发明人不履行自己应承担的义务，就得不到专利权，常常因为发明人没有提供出一份足以实施发明的充分说明书（专利术语叫“可实现的”披露）而由法院判定专利无效。

通常把“专利”作为名词来表示发明人拥有的权利。其法律文件实际被称之为“公开证书 (Letters Patent)”，意指公开给公众的书面文件 (Patent 为形容词，源于拉丁字 patens，是 patere 的现在分词，意指“开放的”“敞开的”)，它与“密封证书” (Letters Close) 即秘密或加有封印的证书相反。美国专利证书的官方文本是一个精致的，饰有彩色缎带并加盖封印的法律文件。

对于专利，有一种最常见的错误概念是，当专利被批准后，申请人便有实施其发明的权利，实际情况往往并非如此。专利所给予专利拥有者的只是排除他人实施该发明的权利。一般来说，一件发明常常是对一个较广泛的发明的改良，而后一发明则是其他人可掌握的另一一些“优势”发明的主题。某项专利产品的制造可能需要使用别人已获专利的工艺过程。其他人的专利也可能包括某项专利产品的各种重要应用。想实施专利的错误打算可能会和反垄断法冲突。所有这些因素必须在实际使用专利时加以考虑。这些问题在第九章和第十章中将进一步讨论。

专利制度着重考虑的是公众利益，法院反驳专利有效性的决定，通常是根据专利权人是否对知识真正做出了一个新的、非显而易见的和有用的贡献，同时最起码也是较重要的一点则是要看看对于新发明的披露是否直接而全面。美国专利和商标局在审查专利申请时的职能是双重的：(1)防止由实际并没有做出创造性贡献的申请人不公正地抢先占有已知

的技术或显而易见的技术；（2）鼓励披露发明以促进技术的不断发展。美国专利和商标局在竞争力量和社会合作的需要这二者之间建立平衡所取得的成功，确实已经成为美国技术优势的一个强大推动力。

第二章 如何阅读专利文献

专利文献是具有一定文体和格式的文件。许多化学家和化学工程师对之采取敬而远之的态度，因为他们对于专利文献所用的语言和结构均感陌生，从而觉得使用不便。再者，还可能是他们，尤其是学术界，普遍认为专利不是一种十分确切可靠的技术情报源。如果懂得专利文件的结构，弄清它之所以如此书写的道理，那么就会使人感到有效地使用这一重要情报资源并非难事。

专利不同于科技论文，后者要求读者具备一定的基础知识，而专利则必须依靠本身的内容。专利不考虑读者现有的知识水平如何，而只要求读者掌握跟专利有关的知识中的一般技艺。

每件专利都是就一项单独的问题进行阐述，内容包括：问题本身，问题的解决方案，以及发明人针对其发明项目提出的、涉及制造细节和实际应用的种种可能。因此，一件专利，其特点是，既有事实的资料，又有推理的内容，而读者凭自己的经验，便可对专利所披露的这两个方面的内容予以区别、挑选和评价。不过，阅读专利必须遵循一定的方式方法，与阅读科技论文全然不同。

专利的内容

每件专利所披露的内容几乎都包含一些共同的要素，通常其顺序如下：

- (1) 对某个技术领域的陈述——即主题。

(2) 对现有技术水平的讨论——即背景情况和说明需要解决的问题。

(3) 发明“目的”的陈述——即发明所带来的好处。

(4) 发明的简述或“定义”——即用科技术语表达发明所提问题的解决方案。

(5) 在简述中扼要地概括发明在各方面的细节。

(6) 对发明用途(“实用性”)的叙述。

(7) 实施举例。

(8) 权项, 即从法律角度叙述批准授予发明人独占权的内容。

现代的美国专利一般是明确地分成几个部分, 概括了上述各项要素的全部内容。早些年的美国专利以及许多其他国家的专利则可能不会象目前这样清楚明确, 但每个要素总要以某种程度在公开内容或权项中提到。

下面是美国专利 3987074 号的实例, 有些专利比这短, 而有些则更长, 据知, 有一件电子学方面的美国专利, 其说明书竟长达900页以上, 是迄今最高纪录保持者。

United States Patent (19)		(11) 3,987,074
Haase et al.		(43) Oct. 19, 1976
[54] PROCESS FOR THE MANUFACTURE OF VANADYL ALCOHOLATES	3,657,293 4/1972 McCoy et al. 260/429 R	
[75] Inventors: Ranier Haase, Bokel (Oldenburg); Arnold Lenz, Cologne-Stammheim, both of Germany	1,271,641 1/1962 France 1,816,386 2/1970 Germany	FOREIGN PATENTS OR APPLICATIONS
[73] Assignee: Dynamit Nobel Aktiengesellschaft, Troisdorf, Germany		OTHER PUBLICATIONS
[12] Filed: Aug. 21, 1974		Bull. Acad. Sci. USSR pp. 899-900 (1957).
[11] Appl. No.: 499,251		Primary Examiner—Helen M. S. Sneed Attorney, Agent, or Firm—Burgess, Dieklage & Sprung
[30] Foreign Application Priority Data Aug. 25, 1973 Germany 2345056 Dec. 17, 1973 Germany 2365704		[57] ABSTRACT
[52] U.S. Cl. 260/429 R; 252/431 R		A process for the preparation of a vanadyl alcoholate which comprises contacting vanadium pentoxide with an alcohol in the presence of an orthoester of the formula R ¹ C(OR ²) ₃ wherein:
[51] Int. Cl. C07F 9/00		R ¹ is hydrogen, a straight-chained alkyl group of 1 to 5 carbon atoms or a branched chain alkyl group of 1 to 5 carbon atoms; and
[58] Field of Search 260/429 R		R ² is a straight-chained alkyl group of 1 to 12 carbon atoms, a branched-chain alkyl group of 1 to 12 carbon atoms or phenyl.
[56] References Cited		12 Claims, No Drawings
U.S. Pat. 3,432,445 3/1969 Asgan 260/429 R X		
3,455,974 7/1969 Lion 260/429 R		
3,652,617 3/1972 Termin et al. 260/429 R		

PROCESS FOR THE MANUFACTURE OF VANADYL ALCOHOLATES

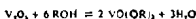
BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a process for the preparation of vanadyl alcoholates, particularly vanadyl alcoholates, of the formula: $O = V(OR)_2(Y)_n$, wherein Y is $\equiv V=O$ and n is 0 or 1 and when n is 0, R is alkyl, cycloalkyl, arylalkyl, arylalkyl, aryl, alkoxyaryl or hydroxyalkyl and when n is 1, R is an alkoxy radical. This invention is particularly addressed to the problem of removing water formed during the preparation of vanadyl alcoholates by reaction of vanadyl pentoxide with an alcohol. This invention is also directed to the preparation of vanadyl alcoholates of high purity.

2. Description of the Prior Art

It is known that vanadium pentoxide can be reacted with alcohols to form the corresponding vanadyl alcoholates according to the following reaction:



Since this reaction is an equilibrium reaction it is necessary, in order to favor the formation of esters, to continually remove the water that forms. The continual removal of the water, however, involves great difficulty.

It is known to remove the water by distilling over the water as it forms in the reaction, together with excess alcohol, into a second reaction vessel containing a substance which absorbs water, such as quicklime for example. At the boiling temperature of the alcohol, and with intensive stirring, the alcohol is dewatered in the second reaction vessel and then distilled back into the first reaction vessel in which the reaction with vanadium pentoxide is taking place. (Cf. German Offenlegungsschrift 1,816,386).

This known process, however, is very expensive. For example, the water-removing substance must be constantly replaced or regenerated. Also, constant vigilance is necessary during the reaction to prevent the reaction from stopping prematurely due to the exhaustion of the water-removing substance. Heat must continually be introduced into the mixture captured in the second reaction vessel in order to keep it boiling. This relatively great energy consumption is particularly appreciable when operating on a commercial scale.

Another disadvantage of this known process consists in the fact that it is necessary to operate in the presence of a strongly acid catalyst. Despite the presence of the catalysts, however, it is necessary to reflux the reaction mixture for more than 8 hours as a rule if it is desired to achieve pure ester yields of about 50%. Yields greater than 50% can be achieved only when operating on a laboratory scale. When the procedure is used on a commercial scale it is found that the yields vary from batch to batch. Consequently, the prior-art process does not achieve reproducible results to a sufficient extent.

Operating in the presence of strongly acid catalysts results in additional disadvantages. The amounts of acid added accelerate the reaction of the V_2O_5 with the alcohol to form the ester, and yet they promote the reduction of the pentavalent vanadium to tetravalent vanadium with the cooperation of the corresponding alcohol. The side reaction, however, is not desired.

Since the compounds of the strongly acid reaction are used in practice preferably in amounts of 1 to 10 weight percent with reference to the input vanadium, they are not present in only catalytically effective amounts. Rather, they are present in appreciable percentages which constitutes an impurity in the reaction mixture product. Thus, sulfuric acid is usually found in the form of vanadyl sulfate, in which the vanadium is in the oxidative tetravalent stage. Thus, for example, in a solution of vanadium oxytriisopropylate in isopropanol, with a moisture content of 0.2 weight percent the main part of the tetravalent vanadium is found in the form of solid vanadyl sulfate in the unreacted vanadium pentoxide. In the case of higher moisture contents, the vanadyl sulfate is contained increasingly in the reaction mixture and interferes with the processing.

For example, out of 200 g of V_2O_5 , 3000 ml. of isopropanol and 10 ml. of concentrated sulfuric acid, one obtains after 3 hours of reaction solution which still contains 0.39 weight percent of water. When the unreacted V_2O_5 is filtered out of the solution and the isopropanol has been removed by evaporation and the ester has been vacuum distilled, 143 g (= 26% yield) of pure vanadium oxytriisopropylate is obtained. An extraordinarily great quantity of 109 g of distillation residue remains as an unwanted by-product probably due to the high percentage of sulfuric acid and the excessively great moisture content of the reaction solution.

If organic sulfonic acids, such as toluenesulfonic acid and benzenesulfonic acid, are used instead of sulfuric acid, similar phenomena occur. In the preparation of vanadium oxytriisopropylate, voluminous greenish flakes precipitate from the greenish reaction solution during the progressive concentration and the distillative refinement of the ester that follows; these flakes interfere with the distillation and increase the percentage of distillation residue.

Attempts have already been made to use phenol derivatives or weak acids such as boric acid, for example, as catalysts instead of the strong acids. In the reaction of alcohols having up to 4 carbon atoms, however, these catalysts display no activity (French Pat. 1,271,641).

It is also known in the preparation of vanadyl alcoholates to reflux stoichiometric amounts of vanadium pentoxide and an alcohol having 5 to 5 carbons, respectively, in the presence of benzene (with a ratio of alcohol to benzene of 1 : 1.1 to 1 : 1.35 by volume). The water formed at the boiling temperature is removed from the reaction zone as an azeotropic mixture by the addition of benzene and can then be separated as the heavy phase in a water separator.

At a reaction time of 8 to 12 hours the yields amount to only between 10 and 32% with reference to the vanadium pentoxide input. Especially in the preparation of vanadium oxytri-n-butylate, yields of only 26%, for example, have been achieved (cf. Bull. Acad. Sci. USSR 1957, pages 899-900).

In the preparation of vanadium oxytri-n-butylate it has also been proposed to add toluene instead of benzene to the reaction mixture as an extractant in order to increase the yield. Yields of up to about 65% can be achieved by this process (cf. U.S. Pat. No. 3,657,295), but the time required for the reaction is very long, amounting to as much as 24 hours. In this process a mixture of vanadium pentoxide, toluene and n-butanol is heated to ebullition and the water forming in the reaction is distilled together with the toluene as an

azeotropic mixture into a water separator. Here a water-rich phase and a hydrocarbon-rich phase are formed. The latter is continuously recycled to the reactor and the water-rich phase is separated at intervals. Using 1.3 to 1.4 times the stoichiometrically required amount of alcohol, the highest yields are achieved in this case, of 65%. According to this patent, the use of a greater excess of alcohol will not achieve any technical effect as regards increasing the yield, increasing the reaction speed, or the like.

The long reaction time (24 hr.) that is required if it is desired to achieve yields of 65% is particularly disadvantageous. Another disadvantage is that very large amounts of toluene must be used (the ratio of toluene to n-butanol is preferably to be from 1 : 1 to 3 : 1 by volume). In the processing of the reaction mixture that follows, the excess solvent — in this case unreacted alcohol together with toluene — is removed by distillation. It has been found disadvantageous that, before the disillate can be used for the next batch, it must first be readjusted to a specific toluene-butanol content, because otherwise reproducible results cannot be achieved. For this purpose, however, complicated procedures are necessary, which make this process uneconomical and render technical scale operation difficult.

To avoid these difficulties in the prior art methods based on the reaction of V_2O_5 with alcohols it has also been proposed that vanadyl alcoholates be prepared by starting out with $VOCl_3$ and reacting it with alkali metal alcoholates or alcohols; this results in yields of 60% (with reference to $VOCl_3$). These known methods, however, have the disadvantage that the reactant $VOCl_3$, which is very sensitive to hydrolysis, has to be prepared in a separate procedure from V_2O_5 . Furthermore, undesired vanadium-containing by-products form in this process, as well as alkali chlorides or HCl as reaction products. The hydrochloric acid that forms has to be neutralized with ammonia in an another separate procedure, so that, by and large, the process is a very complicated one. In addition, these prior-art methods result in products which are not entirely chloride-free. The esters prepared in this manner usually have a reduced shelf life, which is indicated by a dark discoloration. Often they are then no longer suitable for use as a component of a polymerization catalyst.

The preparation of vanadyl alcoholates of C_2 to C_4 alcohols by the transesterification of a lower vanadyl alcoholate with a correspondingly higher boiling alcohol is relatively difficult. These transesterification methods require that the starting product be a vanadyl alcoholate of a low alcohol which in turn is supposed to be more easily accessible than the desired vanadyl alcoholate of a higher alcohol. For the preparation of such "low" alcoholates, however, a process like the one described above (on the basis of $VOCl_3$) has hitherto been recommended. In the transesterification processes, losses of yield have always had to be accepted in order to prepare pure esters, so that the problem of manufacturing pure esters with a high space-time yield, by the use of a very simple method, has basically not yet been solved.

It is, therefore, an object of the present invention to provide a process for the preparation of vanadyl alcoholates starting with vanadyl pentoxide. It is a particular object of the present invention to prepare vanadyl alcoholates in high purity in good yields within a commercially feasible period of time and especially without the use of separate reaction zones, recycling proce-

dures and the like. It is a particular object of the present invention to provide a process by which those vanadyl alcoholates which have heretofore proved difficult to prepare can be synthesized in high purity and within commercially feasible period of time with respect to space-time yield.

SUMMARY OF THE INVENTION

The objects of the present invention are provided by a process for the preparation of a vanadyl alcoholate which process comprises contacting vanadium pentoxide with an alcohol in the presence of an orthoester of the formula $R' \cdot C(OR'')_3$, wherein

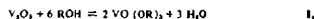
R' is hydrogen, a straight-chained alkyl group of 1 to 5 carbon atoms or a branched chain alkyl group of 1 to 5 carbon atoms; and

R'' is a straight-chained alkyl group of 1 to 12 carbon atoms, a branched-chain alkyl group of 1 to 12 carbon atoms or phenyl.

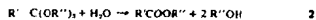
The present invention can be considered to be an improvement over the art known process for the preparation of vanadyl alcoholates wherein vanadium pentoxide is reacted with an alcohol. The improvement comprises including in the reaction mixture an orthoester of the formula $R' \cdot C(OR'')_3$, wherein R' and R'' have the previous assigned significance.

It has been found, in accordance with the present invention, that if an orthoester of such formula is included in a reaction mixture containing vanadium pentoxide and an alcohol that the water formed during the reaction is taken up by the orthoester, which, in turn, is hydrolyzed to form an ester an alcohol.

The invention can be more readily understood when reference is made to the general equation for the reaction of vanadium pentoxide and an alcohol which equation is as follows:



The alcohol is generally employed during this reaction in at least a stoichiometric amount and preferably it is present in excess. The water produced by the reaction would normally present problems in carrying out the process for the water must be removed if the reaction is to proceed to high yields. By including an orthoester of the type described in the reaction mixture, the water is immediately taken up by the orthoester, which, in turn, is hydrolyzed to formic acid esters or to the carboxylic acid ester and the corresponding alcohol, as the case may be, in accordance with the following equation:



DESCRIPTION OF SPECIFIC EMBODIMENTS

In a preferred embodiment of the process of the invention, an orthoester is used which upon hydrolysis releases the same alcohol whose vanadyl alcoholate is to be prepared.

Examples of orthoesters are: orthoformic acid esters such as, for example, trimethyl or triethyl orthoformate, triisooamyl orthoformate, tripropyl orthoformate, triphenyl orthoformate and the like, as well as the corresponding orthoesters of acetic acid, propionic acid, butyric acid and the like. In general, these orthoesters are used which are soluble in the particular reaction mixture that is prepared.

The formic acid ester, or the carboxylic acid ester such as acetic acid methyl ester or propionic acid